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**ANALYSIS AND INTERPRETATION OF THE INTERIOR  
PAINTED FINISHES OF THE MATHEWS-LOCKWOOD MANSION**

**Susanna C. Fourie**

**A THESIS**

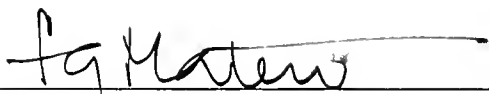
**in**


**Historic Preservation**

**Presented to the Faculties of the University of Pennsylvania in Partial Fulfillment of  
the Requirement for the Degree of**

**MASTER OF SCIENCE**

**1997**

  
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Fine Arts/NA/02/1997/F 775

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## **Summary**

### **Problem Focus**

The painted interior decorative surfaces of Lockwood-Mathews Mansion are the subject of this thesis. Research was done on seventeen rooms to determine binders, pigments, and whose designers were not documented

Defining a style to the Lockwood-Mathews Mansion is difficult since it is one of the first designs of its time period. Although in retrospect one might classify it as a French Second Empire country house. Completed in 1868, it is a fifty-two room mansion designed by Detlef Lienau. The interior architectural ornament and integral furnishings rank among the finest examples and are considered among the first of its kind.

The essence of the problem is the analysis and interpretation of the surface finishes in a physical, artistic, and technical context. This includes documentation of the variety and material content of the original architectural fabric in the mansion.

The Music Room has been selected for conservation treatment during the Summer of 1997 and will serve as a training program for students as well. Therefore, the analysis of the thesis focuses on this particular room.



## **Theoretical Development of the Problem**

The recognized approach to material analysis and interpretation is a thorough understanding of the function, techniques, and materials used for the architectural surface. Not only is the surface of aesthetical value, but it forms the skin of the building. The paint layers are the most prone to entropic effects of nature - light, moisture, temperature, and pollution. Interpretation of the paint layer stratigraphy is done diachronically (through-time) and synchronically (at one time). The research and study of the decorative techniques, materials, methods of application, and original color were done to develop a clear understanding of the decorative surfaces. The study includes the analysis of the physical properties and constituencies of the paint layers, i.e., pigments, binders and the substrate.

## **Methodology**

The procedure to answer the stated thesis problem is comprised of six orders:

- 1) A literature survey to gain an understanding of the nineteenth century paint techniques and materials.
- 2) A literature survey of analytical techniques.
- 3) A literature survey of restoration done on the Lockwood-Mathews Mansion.
- 4) Examination of decorative surfaces by taking representative samples in rooms of each of the four designers. After initial sample analysis and interpretation of the cross-sections are completed, a detailed study of the Music Room is done.
- 5) A detailed material analysis is conducted by combining current research with cross-sectional samples identification techniques and confirmatory test methods of XRD, FT-IR and SEM.
- 6) Color notation of colors found in house using the Munsell color chart.





## 1. History of Lockwood-Mathews Mansion

The Lockwood-Mathews Mansion has a rich history. Located in Norwalk, Connecticut, it was built for Le Grand Lockwood, a wealthy broker. The French Second Empire style mansion was considered as the most sumptuous private home in America of its time. The best materials were used and the workmanship was said to be absolutely perfect<sup>1</sup> (Fig. 1).

Detlef Lienau, a Paris trained architect who introduced the mansard roof in the United States in 1850, designed the Lockwood-Mathews Mansion. He combined a classic plan with contemporary European and American features. Two turret-topped towers, a large veranda, and a conservatory satisfied the Victorian demand for the picturesque. Three and a half stories high, the mansion has fifty-two rooms around a central octagonal rotunda soaring forty-two feet to a high double skylight. The floor plan consists of a Greek cross with the corners made by the arms of the cross filled in, bays on the three sides, and the entrance on the fourth (Fig. 2). As befits its fortress-like construction, double exterior walls of closely fitted grey granite ashlar with four inches of insulation air space between them sits on stone foundations three feet thick. The inner brick bearing walls are twelve inches thick, and the roof is covered with slate and tern metal. It is no wonder that the mansion cost almost two million dollars to construct.<sup>2</sup> Twenty years passed before the great Victorian "Cottages" of New York and New Port would match its splendour.<sup>3</sup>

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<sup>1</sup>Bergmann, Richard. *Restoration of the Lockwood-Mathews Mansion Preserving a Masterpiece of Craftsmanship*. Technology and Conservation, Fall 1982, Vol. 7 No. 3 p.14.

<sup>2</sup>Ibid. pg. 14

<sup>3</sup>Adams, Mary. *LeGrand Lockwood 1820-72*. Lockwood -Mathews Mansion Museum of Norwalk, 1960, p.2.



Construction of the house began in 1864.<sup>4</sup> Over 200 masons, stonecutters, woodcarvers, and assorted artists and artisans travelled from Europe to work on the monumental building, arriving on ships that contained rare woods and marble used in the construction. The designers, Leon Marcotte, George Platt, and Christian and Gustave Herter matched the interior architectural features with the carved and inlaid surfaces of the upholstered furniture and cabinetry in woods, design, color, and finish. Pierre Victor Galland, the great decorator of the Second Empire period, collaborated on three rooms with the Herter Brothers. The rooms, each decorated in a different style, are lavishly ornamented with porphyry, marbles, bronze medallions, carved and inlaid wood, and embellished ceilings.<sup>5</sup> The Library and Dining Rooms are in Renaissance Revival style with elements such as pediments and columns, and cartouches, while the Drawing Room is in the Louis XVI Revival mode. The Music Room is almost a pure Second Empire style or neo-Greg with its acroteria on pedimented doorways and light colored woods. Many Empire elements are found such as Greek and Roman motifs. In the Dining Room, three dimensional Renaissance designs are combined.<sup>6</sup> Whereas, in the Music Room, stencil pattern of the acanthus scroll in two dimensional form is found. This scroll is further designed with laurel and pendant leaves. The laurel border is combined with a gold ribbon.<sup>7</sup> (Fig. 3)

On the second floor, elegant bedroom-sitting room combinations and bathrooms are fitted with Italian marble.

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<sup>4</sup>Bergmann, Richard. *Restoration of the Lockwood-Mathews Mansion Preserving a Masterpiece of Craftsmanship*. Technology and Conservation, Fall 1982, Vol. 7 No. 3 p.15.

<sup>5</sup>Ibid. p. 14.

<sup>6</sup>Findlay, Mary. *Interior decoration of the Mathews-Lockwood Mansion*. Columbia University, 1974, p. 90.

<sup>7</sup>Ibid. p. 79.



By 1868, Le Grand Lockwood moved into his nearly completed mansion. Lockwood mortgaged his house on November 5, 1869, due to his losses in the stock market. In its incomplete state, the mansion had a value of \$800,000.<sup>8</sup> In 1872 Prospectus details were published regarding the grounds and outbuildings, which were thought to have been designed by the foremost landscape architect Frederick Law Olmstead. Shortly after, Le Grand Lockwood died of pneumonia. In 1876, Charles Drelin Mathews bought the mansion at auction.<sup>9</sup>

The mansion was a family residence for seventy years, after which it was leased to the City of Norwalk for use as a park. Saved from demolition after a court battle between the city and people of Norwalk, restoration began in 1959 to reverse the effects of years of minimal maintenance. However, returning the mansion to its original elegance has proved to be a complex, time consuming project.<sup>10</sup>

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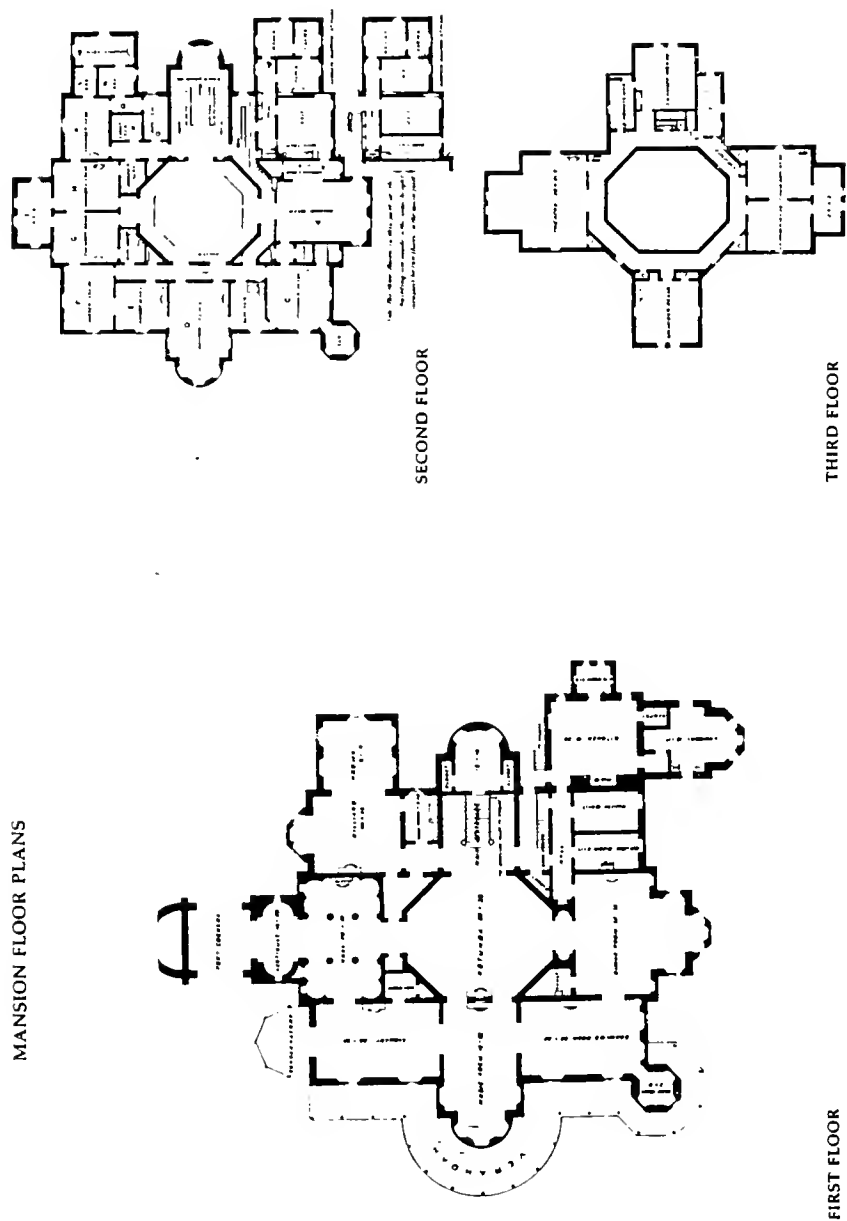
<sup>8</sup>Adams, Mary. *LeGrand Lockwood 1820-72*. Lockwood Mathews Mansion Museum of Norwalk, 1960, p. 2.

<sup>9</sup>Bergmann, Richard. *Restoration of the Lockwood-Mathews Mansion Preserving a Masterpiece of Craftsmanship*. Technology and Conservation, Fall 1982, Vol. 7 No. 3, p.14.

<sup>10</sup>Ibid. p. 15.



Figure 2: Plan of Mansion<sup>11</sup>



<sup>11</sup>Findlay, Mary, and Friend, Doris E. *The Lockwood-Mathews Mansion*. Lockwood-Mathews Mansion Museum of Norwalk, Inc., 1981, p. 18.





**Figure 3: Photograph of Music Room**



## **2. Condition Studies**

### **2.1. Painting Techniques**

It is important to establish a background of the previous restoration done in order to understand the historical decoration techniques and materials that have been identified by previous restorers and to ascertain any reoccurring problems and the reasons behind them.

Through the years, the technique of the wall paintings has been described as “frescoed,” “oil frescoed” and “oil paint.” The Historic American Buildings Survey describes the



wall paintings as oil frescoed.<sup>12</sup> In 1988, Edna Kimbro refers to the paint work done by the Herter Brothers as frescoes,<sup>13</sup> but Morgan Phillips found it to be oil paint.<sup>14</sup>

In 1988 Morgan wrote a report determining whether certain paints and semi-opaque glazes were original or applied after the Lockwood period. He found that all the paints and glazes as well as the gold leaf were original in the dining room.<sup>15</sup> Paint techniques were described by Phillips as follows:

It was found that most elements of the original paint work consist of semi-opaque glazes overlying markedly different (even contrasting) ground colors. Some of the glazes (in the flat fields) were stippeld and some (on the mouldings were brushed). Stippling was commonly used to enhance the effect of matteness, and the difference between fields and mouldings, setting the mouldings off from the fields as if they were made of different materials. Besides the glazed areas (the fields and certain elements of the mouldings), there are elements of the mouldings that are painted (plain brushed) with absolutely opaque plain paints. All these contrasting effects of texture and opacity almost create an impression of a ceiling made of various fine materials rather than plain paint.<sup>16</sup>

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<sup>12</sup>Historic American Buildings Survey HABS No. CONN.-265

<sup>13</sup>Kimbro, Edna E. *The California commissions of the Herter Brothers*. New York. 1988.

<sup>14</sup>Phillips, Morgan. *Letter to Mary Findlay* SPNEA, 2 August 1977.

<sup>15</sup>Phillips, Morgan, Letter Report: *Partial Investigation of Cornices and Ceiling Colors*. SPNEA, Feb. 1988.

<sup>16</sup>*Ibid.*



## 2.2. Paint Deterioration

In the 1995 proposal for SPNEA, Phillips stated that poor paint adhesion of paint to plaster is due to the presence of thin water soluble size at the plaster surface which has been weakened by moisture and allows the strong overlying oil paint to peel (glue size was common treatment for plaster as a preparation for painting oil). He felt that systematic selection of pigments was necessary to match original pigments in terms of historical correctness and resistance to fading.<sup>17</sup>

Mary Findlay analysed the craquelure in 1979. She examined two areas in which the paint curled. Findlay determined that the craquelure stemmed from the presence of moisture in the plaster.<sup>18</sup> In 1984, Morgan Phillips took moisture readings on the 10th and 12th of February. It was done with a Delmhorst BD-7 Moisture Detector in which two sharp prongs are pushed into the wall to measure the dry/wet range. Wet readings in the affected areas were generally higher beneath remaining paint. This indicated that water was coming from behind the paint and trapped against the paint. Areas where no paint peeling occurred registered dry, whereas areas where peeling occurred registered wet. Results show the exterior walls to be wetter than the interior walls.<sup>19</sup>

Phillips concluded that rising damp was not the cause of moisture because the cellar walls and foundation were made of laid granite which would not allow for the upward

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<sup>17</sup>Phillips, Morgan. *Proposal for work 1995, SPNEA*.

<sup>18</sup>Findlay, Mary, E. *Lockwood Mathews Mansion Dining Room Ceiling; An Analysis of Surface Condition and Recommendation for Cleaning*. Jan. 1979, p. 2.

<sup>19</sup>Phillips, Morgan. *Letter to David Byrnes at the Lock wood Mathews Mansion*. SPNEA, 14 Feb. 1984.



movement of moisture. He speculated that condensation might be another reason for the high moisture readings found in the house. Using the psychrometer, Phillips obtained readings of 50 - 55% humidity and a relative humidity of 56% at 64°F. These readings denote the possible occurrence of condensation. However, Phillips did not think that the condensation has been the reason for paint failure. He mentioned also that water was entering through the roof.<sup>20</sup>

Mary Findlay also found black spots on the painted surfaces in the drawing room in 1979. These tiny specks were examined at 60X magnification. In her report, she described the spots as resembling caviar. The sample was treated with a three percent solution of Hydrogen Peroxide. After three minutes, the black spots dissolved completely. A yellow stain was left after removal. Findlay concluded that the spots were organic growth, a kind of mold.<sup>21</sup>

### **2.3. Cleaning**

Cleaning solvents were tested for the removal of a black soot on the wall paintings. Several were tried with no success such as Hexane, Xylene, Methylene Chloride and Acetone. Ammonia and ethyl alcohol were too strong; they dissolved and removed the paint and gold.<sup>22</sup>

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<sup>20</sup>Ibid.

<sup>21</sup>Findlay, Mary, E. *Lockwood Mathews Mansion Dining Room Ceiling; An Analysis of Surface Condition and Recommendation for Cleaning*. Jan. 1979, p. 2.

<sup>22</sup>Ibid. p. 3.





## 2.4. Support Failure

In 1977, Morgan Phillips found that one of the reasons why plaster failure occurs in the Billiard and Dining Rooms is due to the spacing between the laths. The spacing is too wide and does not provide strong keys in the plaster. He found that the plaster breaks away from the lath. Two conditions result from the water damage. The first is that the laths are rotting and second is that the nails which secure the laths are rusting. Another reason that the plaster is delaminating from the supports is that the mouldings are too thick because their weight exceeds the strength of their bond to the lath. The deflection of the floor-ceiling contributes to this problem as well.<sup>23</sup>

In the general technical observation introduction part of his report, Phillips mentions that the cracks in the plaster of the ceiling, cornice and walls of the music and Mrs. Lockwood's rooms are explained in terms of the expansion of the masonry walls (though the exterior walls are faced with granite). All fired clay products expand over time due to hydration reactions with moisture. This is known as moisture-induced expansion or hygric expansion. However, expansion does reach a final limit.<sup>24</sup>

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<sup>23</sup>Phillips, Morgan. *Letter to Mary Findlay*, SPNEA, 2 August 1977.

<sup>24</sup>Phillips, Morgan. *Proposal for work by SPNEA at Lockwood-Mathews Mansion*, Dec 1985, p. 1.



### 3. Historical literature

#### 3.1 Hitchcock List

Henry-Russell Hitchcock's *American Architectural Books* was originally published in 1946. Most of the information found in the Hitchcock list is literature on nineteenth century pigments, paints, and related materials. It is a bibliography for American Architecture students, institutions and for people interested in this field. It ranges from 1775 to 1895. It is not only a list of architectural books; it is an inventory of the source books themselves that influenced and shaped the American architectural taste. It includes treatises, histories, builders guides, house pattern books, illustrated books, and commonly used architects' portfolios. The Hitchcock list is a national register of architectural books until 1895.<sup>25</sup>

While a list can never be complete, Hitchcock's is a comprehensive record. Although there is starting date of 1775, there are thirteen books that are American reprints originally printed in England during the 1500s. It is called the "America incunabula." The Hitchcock list has 46 entries on Asher Benjamin alone. Authors of the Classic Revival, Gothic Revival, Neo-Gothic and the Romantic Eclectic like Downing and Davis are also included. Further, since William Bell's book *Carpentry Made Easy* appeared in 1858, more books and guides to building and carpentry became readily available.<sup>26</sup>

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<sup>25</sup>Placzek, A.K. *Hitchcock List*. July 1976, p. vi.

<sup>26</sup>*Ibid.* p. vi.



During the Civil War, architectural publishing came to a standstill; only three titles were listed. After the war, printing of architectural books sharply increased. The height of publishing came in 1882 with no less than twenty-two books listed. This was the time of the great pattern books such as those by W.H. Randall and Sloan. Their books, published by Palliser and Bucknell, began to represent the mighty American Victorian movement towards suburban taste. Such was the flow of published material that the curve of the building production paralleled the output of books.<sup>27</sup>

Hitchcock takes the list to the threshold of the great modern movement. It is a bibliography of the eighteenth and nineteenth centuries which stops just short of the new era.<sup>28</sup>

### **3.2. Literature on Nineteenth Century Colors**

The materials and techniques available to the painters and vanishers who worked in the last half of the eighteenth century were developed by experimenting during the preceding centuries. Very few major changes occurred in the technology of the finishing crafts between 1850 and 1900. It seems that 1880 was a turning point on the house painting color palette. During the last half of the nineteenth century, emphasis lay on personal judgement, tradition and experience. Color and mixing formulas were not specific or standardized which reinforced the need for professional discretion and the tendency of the trade toward exclusivity. Books were usually pocket size indicating that they were used at the work place. After 1880, however, standardization and precision increased, and technical schools and textbooks appeared.<sup>29</sup>

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<sup>27</sup>Ibid.

<sup>28</sup>Ibid.

<sup>29</sup>Alderson, Caroline. *Recreating a 19<sup>th</sup> Century Paint Palette*, APT Vol. XVI No.1, 1984, p. 47.



Caroline Alderson conducted a statistical examination of color science and composition from 1850 to 1924. She made a preliminary inventory of colors cited in period manuals. A comprehensive list of 471 hues using fifty-two pigments were made. Of these, forty-three colors were referred to the most. Flatted interior paint became fashionable in the 1820's, but oil gloss paint was used when durability was required.

The manuals studied in the Hitchcock list affirm Caroline Alderson's findings that the manuals of the 1850-1895 contained few, if any, color mixing recipes. There are no consistent recipes. The same color did not have to contain the same pigments. One reason for this stems from varying shades that a color name could produce in the minds of painters. The color "beige" meant something different for each painter based on his perceptions of the color, the needs of the project, and the chemicals which made up the pigment.

Towards the end of the century, greens, greenish browns and blue-reds increased while the proportion of yellows, greys, and off-whites decreased. Colors cited most frequently during the earlier period were purple, buff, olive, orange, chocolate, drab, fawn, flesh, straw, and violet. In later publications, brown, fawn, chestnut, olive green, bronze-green, French grey, orange, and purple appeared most often, with drab the most popular of all.

Typical exterior colors during the late Victorian era were green, red, brown and ochre. During the third quarter of the nineteenth century, browns and grays, Downing's "earth colors," played a greater role. The variety of colors imitating specific species of natural building materials decreased such as walnut or Portland stone. Yet, imitative colors such as brick or stone remained very popular. Long after the introduction of the more efficient





flat brushes, the round brush was still available. Despite the availability of paint mills, emphasis on the proper use of grinding colors in oils with the muller and stone persisted--one way of resisting the inevitable tide of industrialization.

Economic factors, both cost and durability, played a role in color use and choice. Distemper was used for low priority, non public areas, such as basements or out-buildings and modest private buildings. Whitewash was regarded as the cheapest and crudest wall covering.

Among oil paints that Alderson has studied, cost varied depending on the pigments used. Earth pigments such as yellow ochre, Venetian red, Indian red, sienna, and umber were among the cheapest. White lead, lampblack, chrome green, chrome yellow, and Prussian blue were reasonable. Vermillion (particularly English or Chinese), artificial ultramarine and lake colors were more expensive. Verdigris, indigo, and carmine were among the most costly ingredients. Expensive pigments were used in important rooms or decorative work that did not require large amounts of paint.<sup>30</sup>

### **3.3. History of Paint and Pigments in the 19th Century**

#### **Paint**

The first paint mill was owned by a Boston painter named Thomas Child around the 1700s. It consisted of a granite trough and ball. The paint mills were of the burstone type. Paint was generally produced locally because of the high shipping costs.<sup>31</sup> White

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<sup>30</sup>Ibid. p. 49.

<sup>31</sup>Martens, Charles R. *Technology of Paints, Varnishes and Lacquers*. New York: Von Norstrand Reinhold Company, 1974, p. 5.



lead paint production began in 1804; it was the first paint produced in the United States.<sup>32</sup> Samuel Wetherill & Sons made white lead at the corner of Broad and Chestnut Streets in Philadelphia.<sup>33</sup>

The art of making paint remained in the hands of the painter until the Civil War. Prepared paints made its first appearance in 1858 and came into general use after the Civil War. By the 1880's the use of ready made paints was spreading rapidly.<sup>34</sup> In 1865, D.P. Flinn patented the first attempt to manufacture casein paint (US Patent number 50,068). He used such components as white oxide of zinc, fresh slaked lime, resin, milk, and linseed oil. After several more improvements to the paint in 1885, 1887, and 1896, Regnier's 1924 patent and further improvements by Atwood led to the modern casein "paste-paint." Casein paint is a mixture of a pigment, as talc, and a binder, as dry casein and lime mixed with water.<sup>35</sup> Some sources refer to the base of the paint with contents of white lead, basic lead carbonate, basic lead sulphate, linseed oil, zinc and asbestine (silicate of magnesia).<sup>36</sup> Devoe mentions that substitutes, notably barytes, for lead and zinc and inferior oils were used in casein paints in later years.<sup>37</sup> In 1867, the production of Ready-Mixed paints in the US started<sup>38</sup> and established itself in the 1870's.<sup>39</sup> In the

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<sup>32</sup>Ibid. p. 4.

<sup>33</sup>*Paint and Varnish Making in Philadelphia*. Educational Committee in Philadelphia, 1917, p.20.

<sup>34</sup>*The Colorful Years 1754-1942: The story of a colonial Venture that became an American Institution*. Devoe and Reynolds Co., 1949, p.37.

<sup>35</sup>Mattiello, Joseph J., Ph. D., *Protective and Decorative Coatings: Prepared by a Staff of Specialists under the Editorship of Joseph J. Mattiello, Ph. D. Vice-President and technical Director, Hilo Varnish Corporation, Volume III, Manufacture and Uses, Colloids, Oleoresinous Vehicles and Paints, Water and Emulsion Paints, Lacquers, Printing Inks, Luminescent Paints, and Stains*. New York: John Wiley & Sons, Inc., 1943. p. 461-462, 465.

<sup>36</sup>*Paint and Varnish Making in Philadelphia*. Educational Committee in Philadelphia, 1917, p.11.

<sup>37</sup>*The Colorful Years 1754-1942: The story of a colonial Venture that became an American Institution*. Devoe and Reynolds Co., 1949, p.33

<sup>38</sup>Martens, Charles R. *Technology of Paints, Varnishes and Lacquers*. New York: Von Norstrand Reinhold Company, 1974, p. 6.

<sup>39</sup>*Paint and Varnish Making in Philadelphia*. Educational Committee in Philadelphia, 1917, p.23.



US, the first extensive use of casein was in the early part of the nineteenth century, although use of casein in England and Germany already had been highly developed at the time.<sup>40</sup> The 1930's mark the development of modern casein paints. The Chicago World's Fair of 1933 and 1934, European national trade barriers, and the business depression in the United States have led to a greater consideration of casein paints because of their relatively low cost.<sup>41</sup>

In 1868, Masury spoke of oil based paints in comparison with water based paints.

With as much property may be said that water is the bone of pigments which are used as water colors, as that oil is the base of oil paints. Water would be the better menstruum for paints, if some process could be discovered of rendering water color painting water-proof.<sup>42</sup>

Masury's statement actually was not far from the mark. By the time his words were published, D.P. Flinn had received his patent for casein paint--a water based paint which, as Masury wanted, is water-proof.

## Pigments

By 1857 the following pigments were being manufactured in Philadelphia: white zinc, chrome green, chrome yellow, Chinese and Prussian blue, zinc green--equal to chrome green but less poisonous, and white lead.<sup>43</sup>

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<sup>40</sup>Sutermeister, E., *Casein and its Industrial Applications*, New York, 1927, p. 106.

<sup>41</sup>Mattiello, Joseph J., Ph. D., *Protective and Decorative Coatings: Prepared by a Staff of Specialists under the Editorship of Joseph J. Mattiello, Ph. D. Vice-President and TECHNICAL Director, Hilo Varnish Corporation, Volume III, Manufacture and Uses, Colloids, Oleoresinous Vehicles and Paints, Water and Emulsion Paints, Lacquers, Printing Inks, Luminescent Paints, and Stains*. New York: John Wiley & Sons, Inc., 1943. p. 461-462, 465.

<sup>42</sup>Masury, John W., *How Shall We Paint Our Houses?*, New York: Appleton & Co., p. 137.

<sup>43</sup>Freedley, E.T., *Philadelphia and its Manufacturers*, Philadelphia: 1857, p. 218.



Already by 1809, John Harrison had established Kensington Laboratories which eventually became the Western White Lead Company. This laboratory began with only a small chamber for the manufacture of sulphuric acid; the cost was \$5000. Soon after between 1810 and 1812, Harrison began producing white lead, red lead, litharge and orange mineral.<sup>44</sup> Lamp black was first produced in Philadelphia. Controversy exists over who first initiated the pigment, but it seems to be Fox of German Town in 1775.<sup>45</sup>

Wetherill Peterson was the first to make Venetian Red in America. The English process for making this pigment could not be used because bituminous coal was used there and only anthracite coal was available in Philadelphia. Instead, Peterson produced it by burning copperas to change this green chemical to a bright red.<sup>46</sup>

Lewas Lehigh Zinc Co. at Bethlehem, Pennsylvania, produced oxide of zinc. Samuel Wetherill developed this method. It was manufactured at Friedenville near the Zinc mine.<sup>47</sup> American Zinc is the same as French Zinc in terms of chemical content, but a difference exists between their physical properties, fineness, whiteness, etc.<sup>48</sup> F.A. Kraft and F. Chase received patents for the use of zinc pigments to coat metals in 1868. Kraft developed "zincing iron" while Chase developed "zincing and tinning bath." Both men were from Philadelphia.<sup>49</sup>

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<sup>44</sup>*Paint and Varnish Making in Philadelphia*. Educational Committee in Philadelphia, 1917, p. 20.

<sup>45</sup>*Ibid.* p. 21.

<sup>46</sup>*Ibid.* p. 21.

<sup>47</sup>*Ibid.* p. 23.

<sup>48</sup>*Ibid.* p. 6.

<sup>49</sup>Downs, Arthur Channing, *Zinc for Paint and Architectural Use in the 19th Century*, APT, Vol. VIII No. 4, 1976, p. 97.





Chrome green is a mixture of chrome yellow (lead chromate) and Prussian blue (ferric ferrocyanide) which are obtained from the metals, chromium, lead, and iron, plus potassium or sodium. Chrome Yellow is made by dissolving a litharge in acetic acid while simultaneously dissolving bichromate of potassium in water. Both are combined together and the mixture of lead acetate and bichromate solution precipitates chrome yellow. The manufacturing of Prussian blue is done by dissolving prussiate of potash in water and concomitantly, sulphate of iron (copperas) in water. Combined together, white ferric ferrocyanide precipitates. Mixed with sulphuric acid and chloride of lime, the precipitate oxidizes to a dark shade of blue.<sup>50</sup>

Alizarin crimson is an organic product made from anthracene, a coal tar derivative. It was the only synthetic organic pigment universally approved for artists' use from its introduction in 1868 to the late 1930's. Considered permanent, alizarins absorb much oil and are slow driers. They were made in a limited range of shades from rosy scarlet to maroon, have a characteristic bluish undertone, and are clear and transparent. Alizarins will not fade from exposure to normal daylight, but some samples show a tendency to become deeper in shade. They can be mixed indiscriminately with all other permanent colors.<sup>51</sup>

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<sup>50</sup>*Paint and Varnish Making in Philadelphia*. Educational Committee in Philadelphia, 1917, p. 7-8.

<sup>51</sup>Mayer, Ralph. *The Artist Handbook of Materials and Techniques*, Revised and Expanded edition by Steven Sheenan, Fifth edition, Faber and Faber, Boston, 1991, p. 94.



### 1. Paint and Decorative Finishes

Paint has two primary functions, that is, to decorate and protect. Color, gloss, texture, or a combination of these, produces decorative effects. As a result of these properties, the reflectance of light is effected. Paint consist of pigment particles dispersed in a medium called a binder. When the paint is applied on a surface, a paint film is formed. During this process the binder dries and the solvent evaporates.<sup>52</sup>

The following section describes materials and techniques such as paint, binders, pigments, glazes and gold leaf. The materials and techniques are discussed because they relate to the results of the analyses in Chapter 3.

### 2. Paint Layer and Techniques

Paint consists of finely divided pigments particles evenly dispersed in a liquid medium or vehicle; it has the property of drying to form a continuous, adherent film when applied to a surface for decorative or protective purposes.<sup>53</sup> Surfaces may be colored or decorated by applying the pigment directly. In pastel painting the protective function may be supplied by a fixative; the application of which is separate from the decorative or color application. And, in fresco, the ground itself supplies the adhesive or binding property. However, paint, in the commonly accepted meaning of the term, usually implies a material that combines these functions - as the typical oil or tempera paint.

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<sup>52</sup>Martens, Charles R. *Technology of Paints, Varnishes and Lacquers*. New York: Von Norstrand Reinhold Company, 1974, p. 2.

<sup>53</sup>Gettens, Rutherford and Stout, George L. *Painting Materials: a Short Encyclopaedia*, Dover, New York, 1966, p. 331.



A paint is made by compounding pigments (powdered colors) with a liquid which is called the vehicle or carrier of the color. Many elements contribute to the degree of ease or difficulty with which a paint may be manipulated or controlled; no good paint is made by simply mixing pigments and vehicle.<sup>54</sup>

Tempera paint films are adequately strong and durable, but when dry, the volume of binder in relation to the volume of pigment is less than that of oil paints. This is because the bulk of tempera is water; and when the paint has dried, a relatively small volume of solid matter remains to bind the pigment particles together. On the other hand, pure oil paint loses nothing by evaporation and normally has a surplus of oil beyond the amount necessary to bind the paint. The binder in tempera completely surrounds the pigment particles, yet there is little or no surplus medium. The surface has a mat or semimat finish, and the layer is porous.<sup>55</sup>

Distemper is an aqueous paint made with a simple glue-size or casein binder. This term is not in common use in the United States. American products under this heading are calcimine and cold-water paints.<sup>56</sup>

## **2.1. Binder of Paint**

Medium is the word usually applied to the binding material or vehicle that holds together pigments particles in paint. A binder of a paint performs the following four functions:

1. Practical: It allows the colors to be applied and spread out.
2. Binding: It locks the pigment particles into a film, protecting them from atmospheric

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<sup>54</sup>Ibid.

<sup>55</sup>Ibid.

<sup>56</sup>Ibid. p. 17.



or accidental mechanical forces and from being disturbed by the application of subsequent coats of paint.

3. Adhesive: It dries and acts as an adhesive, attaching the colors to the ground.
4. Optical: It has an optical effect, bringing out depth and tone of the pigment, and giving it a quality different from that which it possessed in the dry state.<sup>57</sup>

Emulsifying agents can be divided into inert and active classes. Substances which are inert include glue, casein (containing no free alkali), oleates of lead and alumina, stearate of alumina, turpentine, alcohol, glycerine, and starch. Relatively active agents include chloride of lime, sulphate of zinc, silicate of soda, carbonate of soda, caustic soda, lead acetate, borax, and phosphate of soda.<sup>58</sup>

### 2.1.1. Casein

#### Chemistry of Casein

Casein has been established as a complex compound produced by the conjugation of amino acids.<sup>59</sup> <sup>60</sup> Casein is insoluble in water, particularly at its isoelectric point (pH=4.6). It is also insoluble in alcohol and ether. The addition of formaldehyde to casein strengthens the paint.<sup>61</sup> Casein based paints consist of powdered casein, hydrated

<sup>57</sup>Ibid. p. 35.

<sup>58</sup>Holley, C.D. *An analysis of Paint Vehicles, Japans and Varnishes*. New York, John Wiley and Sons, Inc. 1920, p. 110.

<sup>59</sup>Mattiello, Joseph J., Ph. D., *Protective and Decorative Coatings: Prepared by a Staff of Specialists under the Editorship of Joseph J. Mattiello, Ph. D. Vice-President and technical Director, Hilo Varnish Corporation, Volume III, Manufacture and Uses, Colloids, Oleoresinous Vehicles and Paints, Water and Emulsion Paints, Lacquers, Printing Inks, Luminescent Paints, and Stains*. New York: John Wiley & Sons, Inc., 1943. p. 461.

<sup>60</sup>Gettens, Rutherford and Stout, George L. *Painting Materials: a Short Encyclopaedia*, Dover, New York, 1966, p. 6.

<sup>61</sup>Mattiello, Joseph J., Ph. D., *Protective and Decorative Coatings: Prepared by a Staff of Specialists under the Editorship of Joseph J. Mattiello, Ph. D. Vice-President and technical Director, Hilo Varnish Corporation, Volume III, Manufacture and Uses, Colloids, Oleoresinous Vehicles and Paints, Water and Emulsion Paints, Lacquers, Printing Inks, Luminescent Paints, and Stains*. New York: John Wiley & Sons, Inc., 1943. p. 465.





lime, a preservative, inert and hiding power pigments. In early stages, the casein was no threat to the oil based wall finishes. The use of alkali inhibit the solidification of the calcium caseinate gel, and carbon dioxide caused the containers to blow up. These were a few of the problems in the early stages of casein paints.<sup>62</sup> Today, glycerin-plasticizer, phenols, chlorinated phenols, preservatives, and alkaline flourides are added. To improve washability, oil linseed was added. Increased interest in the development of casein paints to their modern standard began with the Chicago World's Fair. Application characteristics are excellent. It dries rapidly due to its semi-porous structure, and this type of structure enhances the hiding power of parable oil-based finishes containing the same pigmentation. Absence of paint odour stems from the ability of the protein component of the aqueous phase to tie up the oxidation products of the oil phase.<sup>63</sup>

The actual amounts of commercial casein present in casein paints is best obtained by determining the nitrogen content using the Kjeldahl method and then multiplying this nitrogen volume by 7.70. The determination may be made on a five gram sample.<sup>64</sup> Differing methods of the Kjeldahl method exist. Brown states that one percent of nitrogen found multiplied by 6.38 will give the percentage of pure casein in the sample,<sup>65</sup> while according to Holley the amount of glue and casein present can be determined by using 6.37 as the multiplier. About ten grams of sample should be used.<sup>66</sup> However, current techniques using a carbon-nitrogen analyser standardize the Kjeldahl method and decrease the time spent and potential mistakes which occur during this intricate method.

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<sup>62</sup>Von Fisher, William, ed. *Paint and Varnish Technology*. New York: Reinhold Publishing Corporation, 1948, p. 389-390.

<sup>63</sup>*Ibid.* p. 391.

<sup>64</sup>Sutermeister, E. *Casein and its Industrial Applications*. New York, 1927, p. 115.

<sup>65</sup>*Ibid.* p. 281.

<sup>66</sup>Holley, C.D. *An analysis of Paint Vehicles, Japans and Varnishes*. New York, John Wiley and Sons, Inc. 1920, p. 111.



It has been known for centuries that the combination of lime with the curd of skim milk has great cementing properties. The casein of cow's milk is composed of 53% carbon, 7% hydrogen, 16% nitrogen, 22.5% oxygen, 8% sulfur, and 8.5% phosphorus. Usually acetic acid is used to precipitate casein out of milk. Soda, borax, quick lime and water are used to insolubilize casein which need alkali contact. Formaldehyde is added to make a more durable paint and acts as a disinfectant. US patent number 745097 and the German patent number 135.745 are identical casein paint methods.<sup>67</sup>

Casein paints are the forerunners of emulsion paints of today. Emulsion paints are characterised by the fact that the binder is present in a dispersed form in water. In contrast, in a solvent paint, the binder is present in solution form. Because of this inherent physical difference, the formulation and handling of emulsion paints differ from conventional solvent systems.<sup>68</sup>

pH is the logarithm of the reciprocal of the hydrogen ion activity. This measure holds for aqueous systems only and has a scale from 0 (strongly acid) to 14 (strongly alkaline) with the neutral point at 7. A unit change in pH corresponds to a tenfold change in acidity.<sup>69</sup> The pH of a water based paint has profound effect on the physical properties and performance of the system. Casein is only soluble in alkaline range and becomes insoluble at a low pH. High pH ranges will hydrolyze materials such as proteins. If possible, volatile alkali such as ammonia should be used for adjusting the pH.<sup>70</sup>

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<sup>67</sup>Uebele, C.L., *Paint Making and Color Grinding*, New York, 1913. p. 375.

<sup>68</sup>Martens, C.R *Technology of Paints and Varnishes and Lacquers*. Robert E. Krieger Publ. Co., Huntington, New York, 1974, p. 513.

<sup>69</sup>Ibid. p. 520.

<sup>70</sup>Ibid. p. 521.



## 2.2. Size

Size is a thin animal hide glue layer that is applied as preparation for the paint layer. It is superimposed to the finish coat or substrate. It has mainly two functions: sealing the substrate and making the texture of the substrate smooth so that paint will be easier to apply.

Size is a solution of waterglass, glue, or any other substance which chiefly consists of gelatinous matter. Gelatine is a substance obtained from the skin, bones, cartilages, muscles, and membranes of animals. Its most remarkable characteristic, or at least that by which it is detected, is its solubility in hot water, and subsequent conversion into a jelly on cooling. According to analysis, it consists of carbon, in the percentage proportions of 46.88; hydrogen 7.91; oxygen 27.22.<sup>71</sup> When in a state of solution, it may be precipitated by either alcohol or tannin; the former produces the effect by withdrawing the water from its combination; the latter by combining with gelatine to form an insoluble compound which, in all its properties, is analogous to leather.<sup>72</sup>

Masury criticizes the use of animal hide glue in 1868. He recommended that it is not acceptable for first class work.<sup>73</sup>

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<sup>71</sup>Higgins, W. Mulligar, Esq. *The House Painter: or Decorators Companion*. London: Thomas Kelly, 1841, p. 139.

<sup>72</sup>Ibid. p.140.

<sup>73</sup>Masury, John W., *How Shall We Paint Our Houses?*, New York: Appleton & Co., p.115.



### 2.3. Gold Leaf

This decorative method requires the use of metals. Usually gold was used, but if it was too expensive, cheaper metals were used.

Gold is distinguished among metals for its ductility and malleability, and therefore, is particularly suited for manufactured in thin leaves. Gold may, in fact, be beaten into a thin leaf not more than 282,000th part of an inch in thickness, and one grain is made to cover 56.75 square inches. This effect is produced entirely by beating. Silver, platinum, or copper may be reduced to a thin sheet in the same manner. In the production of the gold leaf there are four processes: casting, forging, lamination, and beating.<sup>74</sup>

### 2.4. Glazing

In glazing the colors are thinly mixed, so as to be transparent. This painting technique requires the use of colors which are mixed with the proper vehicles, which usually are either oils, varnish, or goldsize; or, if in distemper, the medium is usually size, beer, milk, etc. This distemper color, when mixed, becomes what sometimes is termed "washing". The purpose of glazing is to deepen the tones of color and to give a warmth or coolness to their hues. By the use of glazing, shadows are made stronger and more prominent, while the warmth or coolness of colors are regulated.<sup>75</sup> As an example for glazing, the effect of gold is created by white lead, golden ochre, vermilioned, and glazed with raw sienna. Old Gold is made by using middle chrome, vermilion, burnt sienna, and glazed with cobalt (thin); or white lead, oxford ochre, and glazed with burnt sienna.<sup>76</sup>

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<sup>74</sup>Higgins, W. Mulligar, Esq. *The House Painter: or Decorators Companion*. London: Thomas Kelly, 1841, p. 142.

<sup>75</sup>Millar, Andrew. *Scumbling and Colour Glazing: A Practical Handbook for House Painters, Coach Painters and Others*. London: The Trade Publishing Co., Ltd., New York: The Painters Magazine, 1909, p. 15.

<sup>76</sup>Ibid. p. 69.





## 2.5. Pigments

A definition for a pigment by the Color Manufacturers Association (DCMA) is as follows:

A pigment are colored, black, white or fluorescent particulate organic and inorganic solids which usually are insoluble in, and essentially physically and chemically unaffected by, the vehicle or substrate in which they are incorporated. They alter appearance by selective absorption and/or by scattering of light. Pigments are usually dispersed in vehicles or substrates for application, as for instance in inks, paints, plastics or other polymeric materials . Pigments retain a crystal or particulate structure throughout the coloration process. As a result of the physical and chemical characteristics of pigments, pigments and dyes differ in their application; when a dye is applied, it penetrates the substrate in a soluble form after which it may or may not become insoluble. When a pigment is used to color or opacity a substrate, the finely divided insoluble solid remains throughout the coloration process.<sup>77</sup>

Physical properties of pigments are those properties inherent to a material itself and which do not dissolve its relationship or combination with other materials. Color is the most important property. A pigment is a finely powdered, colored substance that imparts its color effect to another material either when mixed intimately with it or applied over its surface in a thin layer. When a pigment is mixed or ground in a liquid vehicle to form a paint, it does not dissolve but remains dispersed or suspended in the liquid. Colored substances that dissolve in liquids and impart their color effects to materials by staining or being absorbed are classified as dyes.<sup>78</sup>

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<sup>77</sup>Koleske, V. Joseph, *Paint and Coating Testing Manual*. 14<sup>th</sup> ed. Of the Gardner-Sward Handbook, ASTM Manual Series, Chapter by Peter A. Lewis *Colored Organic Pigments*. Philadelphia, 1995, p.190.

<sup>78</sup>Gettens, Rutherford and Stout, George L. *Painting Materials: a Short Encyclopaedia*, Dover, New York, 1966, p. 143.



Materials used as artists' pigments have requirements other than color. The term "pigment properties" refers to these requirements. Powdered materials that become colorless or virtually colorless in paints are called "inert pigments," a technical term or classification that has no reference to chemical inertness or stability.<sup>79</sup>

### 2.5.1. Classification of Pigments

Pigments used in paints and coatings may be broadly divided into opaque or hiding whites and colored toners.<sup>80</sup> Pigments may be classified according to color, use, permanence, etc. It is customary, however, to classify them according to origin, as follows:

#### 1. Inorganic (mineral)

- a. Native earths: ochre, raw umber, etc.
- b. Calcined native earths: burnt umber, burnt sienna, etc.
- c. Inorganic synthetic colors: cadmium yellow, zinc oxide, etc.

#### 2. Organic

- a. Vegetable: gamboge, indigo, madder
- b. Animal: cochineal, Indian yellow, etc.
- c. Synthetic organic pigment.<sup>81</sup>

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<sup>79</sup>Ibid. p. 112.

<sup>80</sup>Koleske, V. Joseph, *Paint and Coating Testing Manual*. 14<sup>th</sup> ed. Of the Gardner-Sward Handbook, ASTM Manual Series, Chapter by Peter A. Lewis *Colored Organic Pigments*. Philadelphia, 1995, p.191.

<sup>81</sup>Gettens, Rutherford and Stout, George L. *Painting Materials: a Short Encyclopaedia*, Dover, New York, 1966, p. 112.



Broadly speaking, the colored inorganic pigments are either lead chromates, metal oxides, sulfides, or sulfoselenides with few miscellaneous pigments such as cobalt blue, ultramarine blue, iron blue, and bismuth vanadate yellow.<sup>82</sup> Inorganic synthetic colors manufactured with the use of high temperatures are generally of the greatest permanence for all uses, while those requiring delicate or very accurately balanced processing are less so. In general, pigments derived from natural sources such as vegetable pigments are less permanent than the average synthetic color. Many of them are remarkably permanent, but others, particularly the older ones, are fugitive and have the defect of bleeding in oils. Many require the addition of inert bases during manufacturing.<sup>83</sup>

The native earths used as pigments occur all over the world, but there is always some special locality where each is found in superlative form or where conditions have been established which permit its being purified to a greater or more uniform extent than is economically possible elsewhere. Earth pigments are those which are obtained from minerals, ores and sedimentary deposits from the earth's crust. They are those complex mixture of minerals that comprises the clay, ochres, siennas and umbers. Carbonaceous pigments, like Van Dyke Brown, also belong to this group.<sup>84</sup>

### **2.5.2. Inert Pigments**

The inert fillers or extenders are white or nearly white pigments that have low refractive indices and therefore, when ground in oil in the manner of the usual artists color, have little or no opacity or tinctorial effect. They are relatively inexpensive and are easily

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<sup>82</sup>Koleske, V. Joseph, *Paint and Coating Testing Manual*. 14<sup>th</sup> ed. Of the Gardner-Sward Handbook, ASTM Manual Series, Chapter by Peter A. Lewis *Colored Organic Pigments*. Philadelphia, 1995, p. 209.

<sup>83</sup>Gettens, Rutherford and Stout, George L. *Painting Materials: a Short Encyclopaedia*, Dover, New York, 1966, p. 121.

<sup>84</sup>*Ibid.* p. 121.



incorporated in the coating.<sup>85</sup> Most are white or near white inorganic minerals. They are used as cheapeners or adulterants, and to impart to oil paints various properties such as bulk, tooth, reinforcement of the film, hardness, softness, etc. When mixed with aqueous binders or mediums, they are less transparent, and in some cases, as in chalk-glue gesso mixture, several of them will produce brilliant, white, and adequately opaque coatings.<sup>86</sup> The following are the more important commercially available inert pigments: Alumina hydrate, Asbestine, Barytes, Blanc Fix, Chalk, Gypsum, Infusorial earth, Magnesium carbonate, Marble dust, Mica, Pumice, Silica, Talc, and Whiting.

### 2.5.3. Pigments Chemical Properties

Chemical purity of pigments varies greatly. Some are simple, almost pure compounds, while others of equally high quality contain minor components such as natural impurities during manufacture to modify color or pigment properties. Pigments are made from a wide variety of chemical compounds. This explains why they differ greatly in respect to their chemical properties. Among the inorganic coloring materials are the oxides, sulphides, carbonates, chromates, sulphates, phosphates, and silicates of the heavy metals. A very few like Prussian blue and emerald green are complex metallic - inorganic compounds. Carbon in the form of lamp black or charcoal and the metal pigments like gold and aluminium are the only elements that serve in a relative pure state. Dye stuffs are complex organic compounds.<sup>87</sup>

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<sup>85</sup>Koleske, V. Joseph, *Paint and Coating Testing Manual*. 14<sup>th</sup> ed. Of the Gardner-Sward Handbook, ASTM Manual Series, Chapter by Peter A. Lewis *Colored Organic Pigments*. Philadelphia, 1995, p. 217.

<sup>86</sup>Gettens, Rutherford and Stout, George L. *Painting Materials: a Short Encyclopaedia*, Dover, New York, 1966, p. 121.

<sup>87</sup>*Ibid.* p. 138.





## Chapter 3 Sample Analysis and Characterization

### 1. Analytical Methods

This section is concerned with the analytical methods that can be used to identify binders and both inorganic and organic pigments through the use of relatively straightforward and widely accessible techniques such as polarized light microscopy.

Because funds and available instrumentation are often quite limited, low technology methods of analysis are of fundamental importance. Arguably, the most useful of these are the optical microscope and micro-chemical or spot tests. New methods of characterization are being explored such as the identification of organic binders based upon tests developed for the medical industry to identify broad classes of compounds such as proteins, sugars, oils, etc.<sup>88</sup> Additionally, the practical possibilities of applying thin layer chromatography to these problems are becoming increasingly recognized.<sup>89</sup>

The process of identifying pigments using microscopy and micro chemical tests has been described by McCrone<sup>90</sup>, Feller and Bayard<sup>91</sup>. McCrone's work on the identification of the pigments (red iron oxide and vermilion) and binder (glue) in the Shroud of Turin demonstrates how both pigment and binder may be identified through the use of polarized light microscopy and micro chemical tests in conjunction with reconstruction of possible painting processes.<sup>92</sup>

<sup>88</sup>Stulik and Florsheim. *Binding Media Identification in Painted Ethnographic Objects*, JAIC, 30, no. 3, 1992, p. 275-288.

<sup>89</sup>Striegel and Hill. *Methods in Scientific Examination of Works of Art. Vol. I and II*, Technical Report, Getty Conservation Institute, Marina Del Rey, 1994.

<sup>90</sup>McCrone, 1979, 1982, McCrone et al. 1984.

<sup>91</sup>Feller, Robert. *Artists' pigments: A Handbook of Their History and Characteristics*, National Gallery of Art, Washington, Cambridge University Press, Cambridge, 1986.

<sup>92</sup>McCrone, 1980, 1981, 1986, 1987-1988.



New advances in analytical techniques changes the manner in which organic binders in paint are identified and characterized. Masschelein-Kleiner, Tricot Markx, Taets, Pickova, Zelinger, and White provide reviews for general classes of organic materials used as vehicles and binders. Mills and White's 1987 survey of organic materials' structures and chemistry provides a complement to these reviews.<sup>93</sup>

### 1.1. Spot Tests and Micro Chemical Tests

*Spot Tests in Organic Analysis* (Feigl and Oesper 1966) and *Spot Tests in Inorganic Analysis* (Feigl et al. 1972) are standard reference works on spot tests. When spot tests are observed with the aid of a microscope, they are termed micro chemical tests (see also McCrone 1971 and Schramm 1985-86 on ultra microminiaturization of micro chemical tests).<sup>94</sup>

### 1.2. Staining Techniques

Staining is generally done by applying fluorescent stains to cross-sectional samples. Information about broad classes of binding media is obtained from the interaction of the fluorochrome with the layers within the sample. Fluorochromes are reactive dyes which cause certain materials to exhibit fluorescence in a characteristic manner. Certain fluorescent stains are used due to their ability to properly identify the unknown substance without disturbing the material to which it is attached.

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<sup>93</sup>Hansen, Eric F., Walston, Sue, Bishop, Mitchell Hearn, *Matte Paint: Its History and Technology, analysis, Properties, and Conservation Treatment With Special Emphasis on Ethnographic Objects*, A bibliographic Supplement to Art and Archaeology Technical Abstracts, Volume 30, 1993, p. xxxii.

<sup>94</sup>*Ibid.* p. xxxii.



The fluorochromes selected for use were as follows:

- identification of proteins: Fluorescein Isothiocyanate (FITC), Texas Red Isothiocyanate (TRITC) and Cycloheptaamlyso-dansyl chloride complex (DCC-7A).
- identification of lipids: Rhodamine B; 2'7' Dichlorofluorescein (DCF)
- identification of carbohydrates: Triphenyltetrazolium chloride (TTC)

Mills and White in 1978 describe staining tests as supplementary to gas chromatography for organic analysis. In 1987, Wolbers and Landrey reviewed current methods and materials for media characterization on small cross-sectioned samples, including the performance of stains and fluorescent reactive dyes. Derrick et al. in 1993 pointed out problems in the staining of cross sections due to the porosity of some types of paint and subsequent infiltration of the embedding media during the preparation of cross sections.<sup>95</sup> Messinger investigated DCC-7A in 1992, a new fluorochrome specific for proteins which seems to be more successful than all the previous methods.<sup>96</sup>

## **2. Paint**

### **2.1. Sampling**

A total of sixty-four samples were analysed in the Lockwood-Mathews Mansion. They were taken on the first and second floor. Samples were taken in seventeen rooms. With the Music Room as the detailed study.

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<sup>95</sup>Ibid. p. xxxii.

<sup>96</sup>Messinger, John M. *Ultraviolet-Fluorescence Microscopy of Paint Cross Sections: Cycloheptaamylose-Dansyl Chloride Complex as a selective stain*. JAIC 31, 1992, p. 267.



These rooms were:

- First Floor: Rotunda, Library, Music Room, Secondary Staircase, Wash Room.
- Second Floor: Twin Rooms, Hallway, Mrs. Lockwood's Rooms (3), Mr. Lockwood's Rooms (3), Moorish Room and the Italian Suite. (Fig. 4-8)

Some samples were taken from fragments of plaster and paint that are kept in storage at the Lockwood-Mathews Mansion. These were collected from areas where plaster had fallen off such as in the Library and Rotunda.

Most of the samples are of walls, not ceiling, since they were more accessible. Representative samples were taken of the fields in five rooms that had known designers.

These rooms are:

- Leon Marcotte: Library
- Herter Brothers: Music Room and Italian Suite
- George Platt: Moorish Room
- Hutchingson & Son: Twin Rooms (Fig. 4 and 6)

Samples also were taken to determine paint campaigns diachronically (through-time).

A list of the samples is in the Appendix.





Figure 4: First Floor Plan - Rooms Where Samples Were Taken

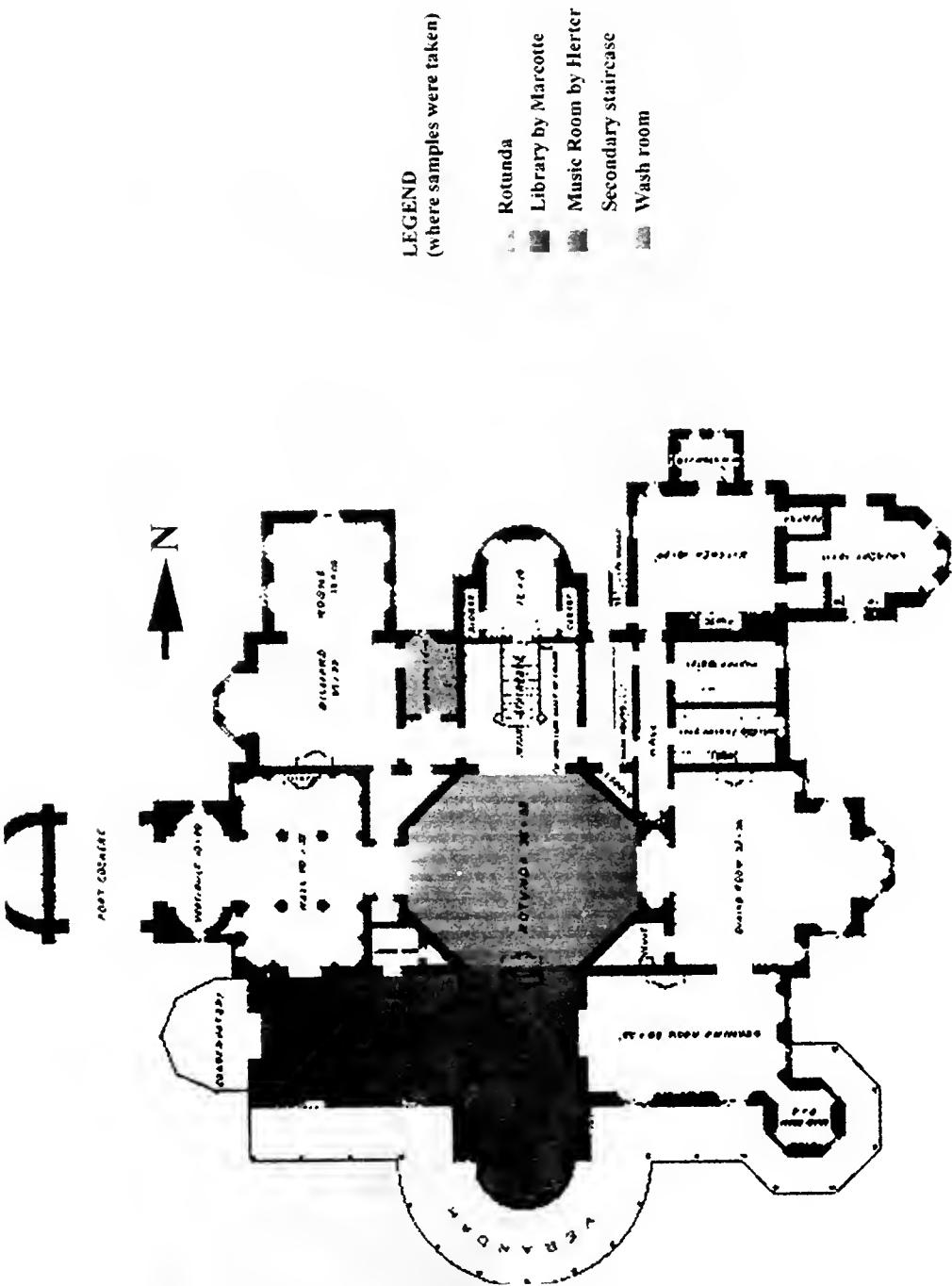




Figure 5: First Floor Plan Showing Location of Samples

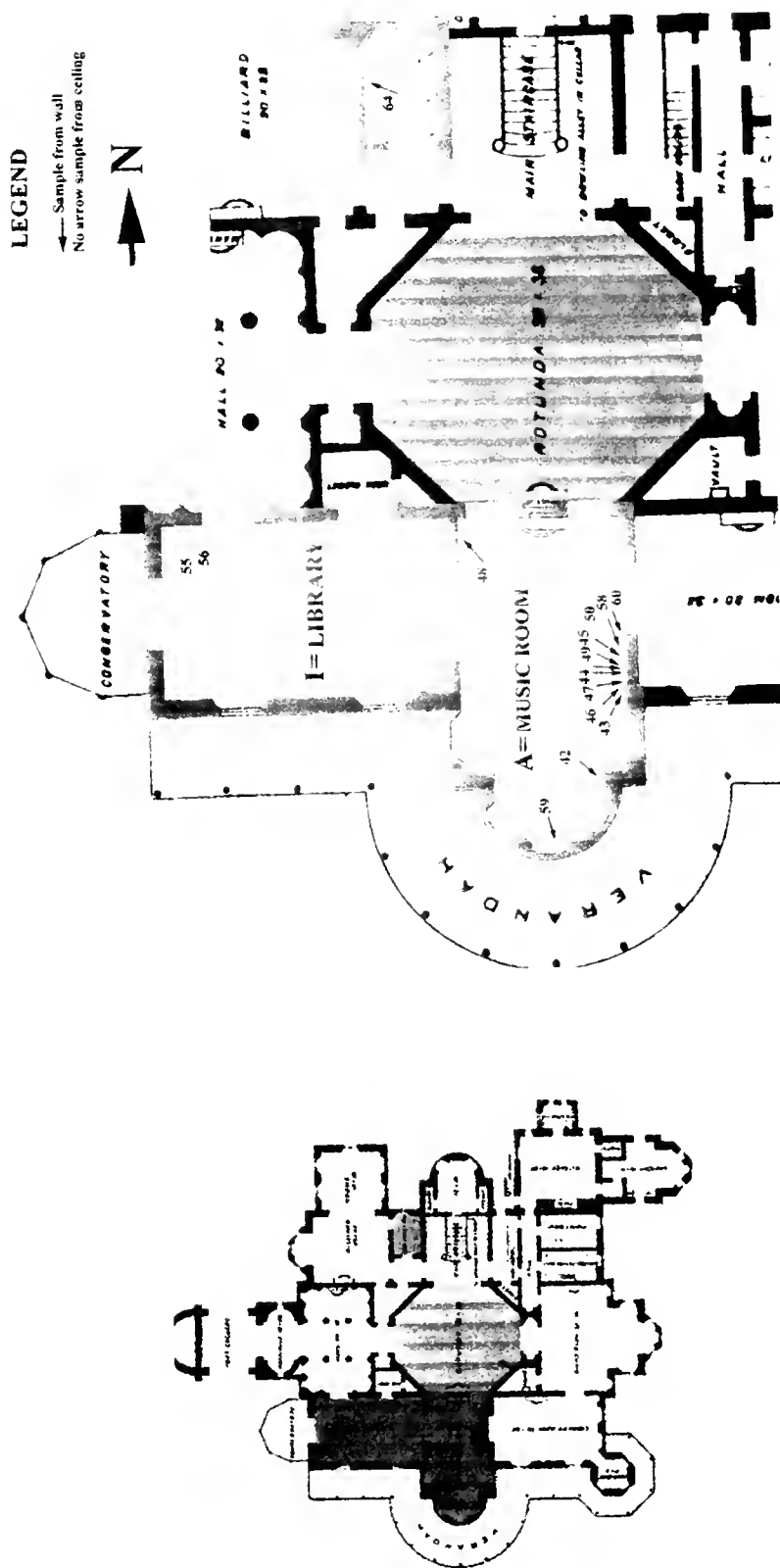
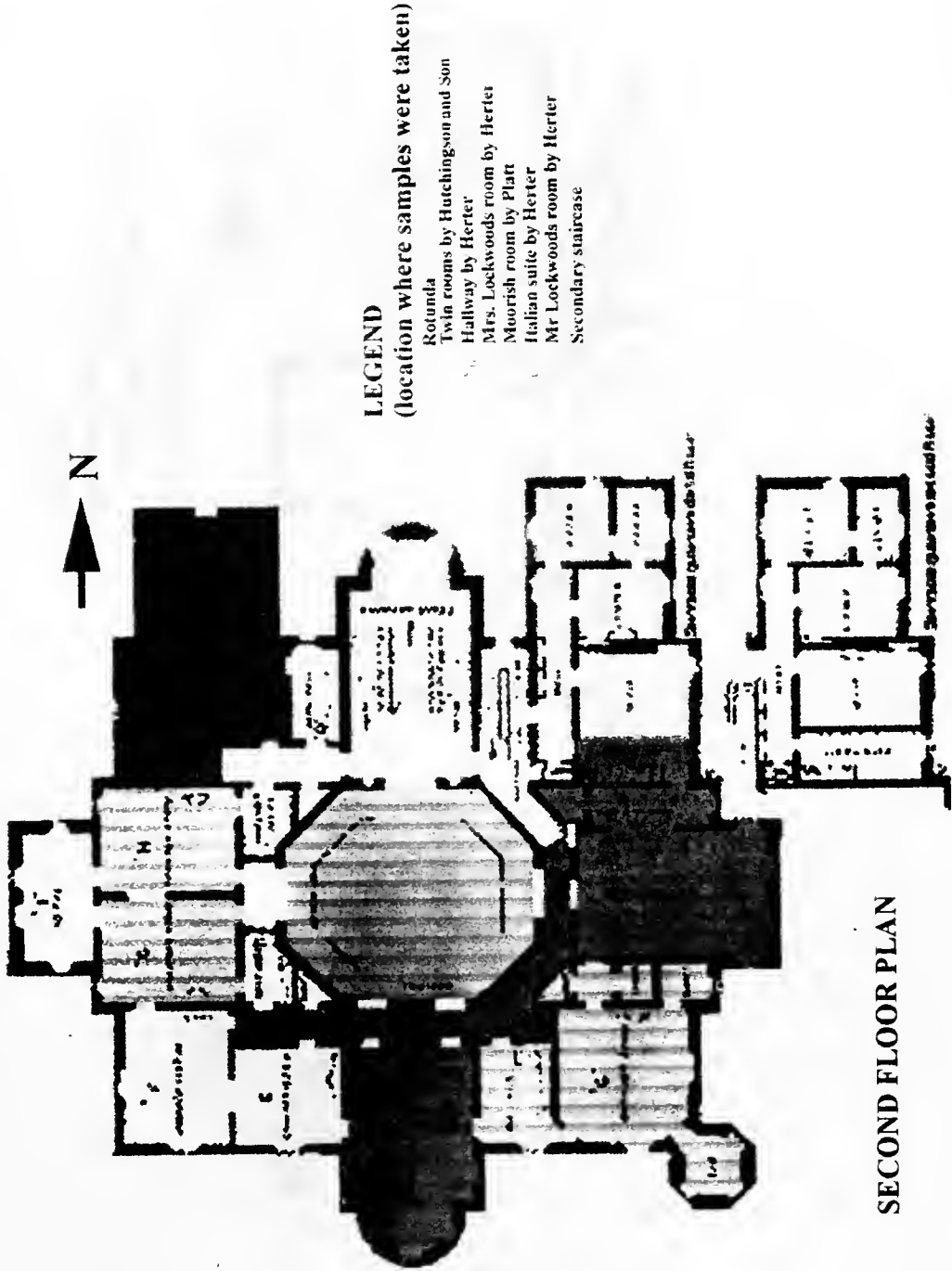




Figure 6: Second Floor Plan- Rooms Where Samples Were Taken















## 2.2. Type of Paint - Binder

Results of fluorochrome analyses with FTIC and FT-IR indicate that casein is the binder of the paint found in the Lockwood-Mathews Mansion. FT-IR revealed that a proteinaceous binder is present. There are two proteinaceous alternatives. They are casein and/or animal hide glue. (Fig. 10) When the paint was tested with fluorochromes, it indicated that there are two proteins present. There is a distinct optical difference between the sizing and the binder of the paint when stained with FTIC. (Fig. 10 - 11) The sizing is brittle and becomes soluble in hot water while the paint is resistant to this test. The paint is remarkably strong as already tested and analyzed by Morgan Phillips and Mary Findlay. Due to this durable property, it was assumed that the paint was oil-based. However, there is no wax or oils present. It was common practice to add glycerine or oil components to the casein to make it more flexible. With Gas Chromatography, the percentage of nitrogen was measured to determine the percentage of binder. The protein value is 0.5518133%. (Fig. 12) Originally, these measurements were obtained by the Kjeldahl method.<sup>97</sup>

A paint, with casein as a binder, consists of a pigment for covering, usually whiting, an inert filler to prevent settling, a pigment for required color, and most important, the insolubilizer constituent. Hydrated lime is used as a insolubilizer. Casein needs to be treated with an alkaline solvent such as ammonia before it can act as a binder.<sup>98</sup> The insolubilizer makes the paint waterproof. The hydrated lime in the paint dissolves the

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<sup>97</sup>Sutermeister, E. *Casein and its Industrial Applications*. New York, 1927, p. 281.

<sup>98</sup>*Ibid.* p. 119



casein when water is added and then forms insoluble calcium-caseinate. The use of ammonia and formaldehyde reacts together as an agent controlling the insolubilizer of casein.<sup>99</sup> The SEM results were suggestive for certain pigments. Certain underlying chemical components were always present in almost every elemental analysis done with the SEM. These elements were interpreted as lithopone,  $\text{CaCO}_3$  and baryite. These chemical compounds are the fillers, inert pigments and the insolubilizer. ( Fig. 13-17)

**Figure 9: Photo Micrograph of Sample 43: Music Room**

Sample no. 43

**Sample Location:** Music Room Field

**Type of Film:** 200 ASA Kodak Royal Gold, Film 5 Negative 11      **Camera:** Nikon

**Magnification:** 225X

**Reflected Light**



Paint Layer with ultramarine pigment particles visible

Sizing

Gypsum substrate

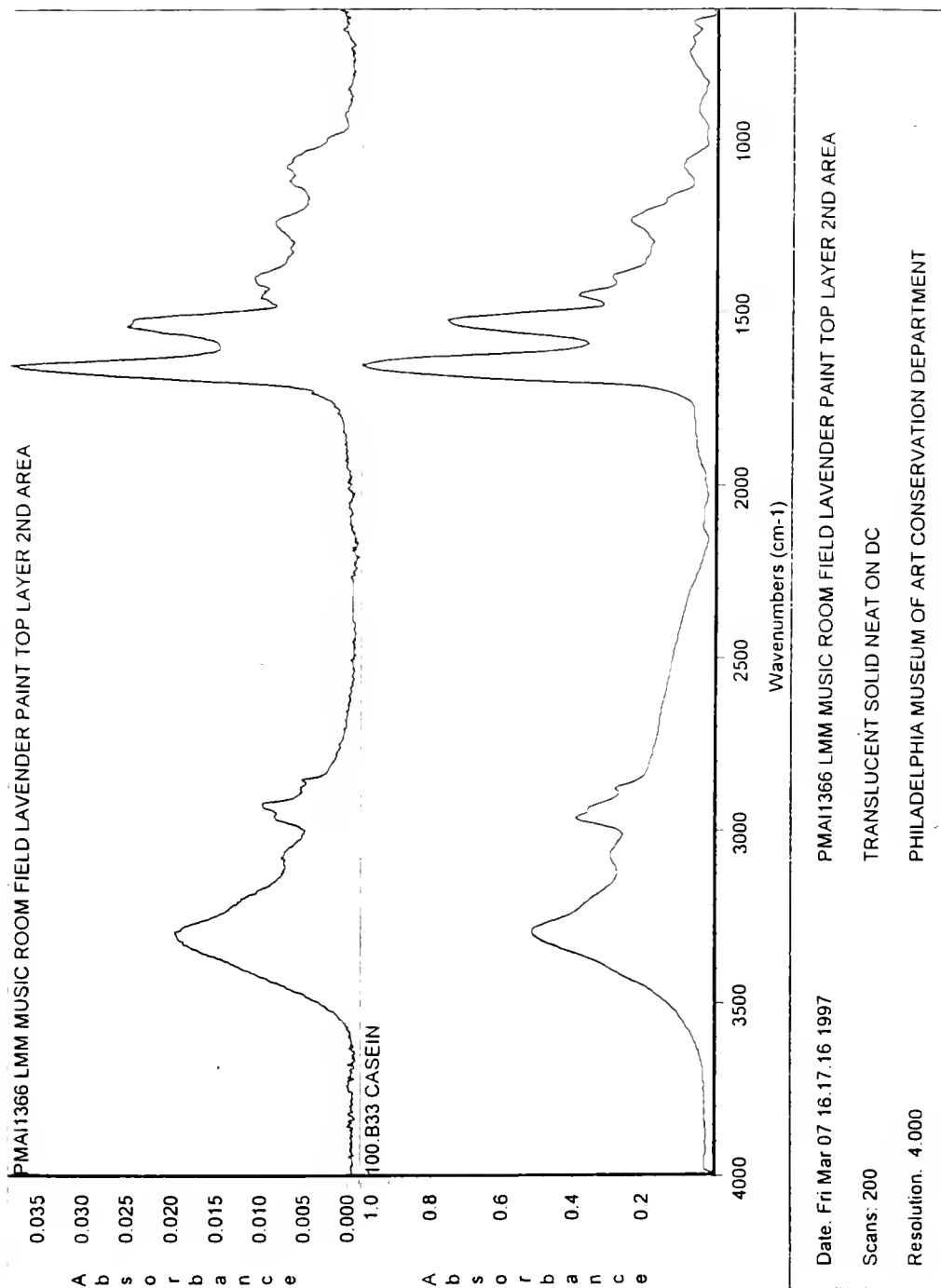
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<sup>99</sup>Ibid. p. 114.



**Figure 10: FT-IR of Paint Determining the Binder.**

Top graph is binder of the paint of sample no. 43 from the field of the Music Room. Indicating a presence of a proteinaceous material. The bottom graph is the standard for casein.







**Figure 11: Photo Micrograph of Sample 50: Music Room - FITC**

Positive for Proteins

Sample no. 50

**Sample Location:** Music Room Field

**Type of Film:** 200ASA Kodak Royal Gold **Before:** Film 3 Negative 20A

**Camera:** Nikon

**After:** Film 4 Negative 10

**Magnification:** 125X

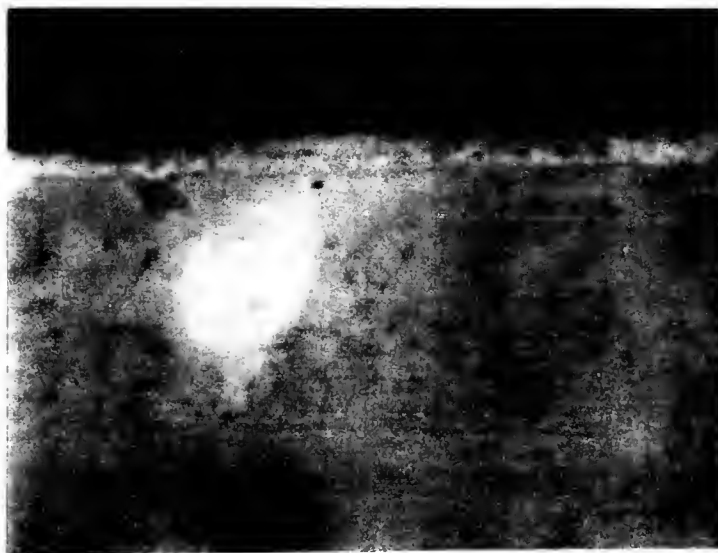
**Before**



Paint Layer, the top brownish layer is part of the design.

Gypsum Substrate

**After**



Paint layer with protein binder, casein.

Gypsum substrate

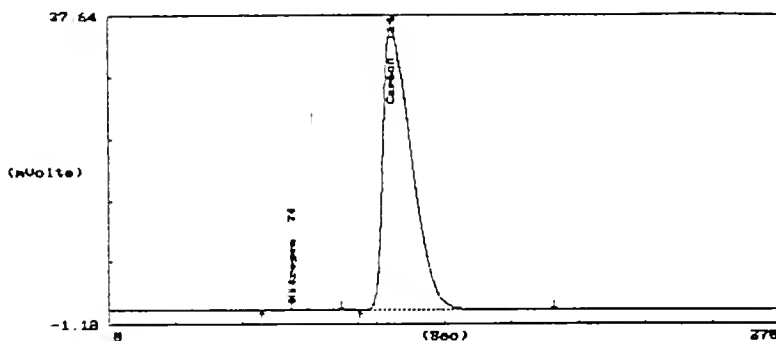


## Figure 12: Gas Chromatography of Sample 59: Music Room

Test for determining the percentage of binder in paint. Sample of Music Room field no. 59 was used. The protein value is 0.5518133%.

### EAGER 200 Stripchart

Sample Ident. : 12 PAINT CHIPS      Filename : RYNTA  
Analysed : 04-02-97 13:44:08      Printed : 04-03-1997 14:08:36



### EAGER 200 Peak Integration Report

Instrument name : Instrument #1      Bline drift (fV): .1  
Company Name : Carlo Erba Instr      Operator Ident. : M.M.D.  
Analysed : 04-02-97 13:44:08      Printed : 04-03-1997 14:08:39  
Sample Ident. : 12 PAINT CHIPS      Filename : RYNTA  
Sample Weight : 5.646      Calc.method: using 'Square to Linear fit'

No. (#)	Type (#)	Start (Sec)	End (Sec)	Ret Time (Sec)	Height (fV)	Area (fV/Sec)	Area % (%)	Name
1	RS	62	94	74	50.7	611	0.18	Nitrogen
2	RS	101	179	114	26184.3	329943	99.82	Carbon
							3305538	100.00

### EAGER 200 Unk Report

Instrument name : Instrument #1      Bline drift (fV): .1  
Company Name : Carlo Erba Instr      Operator Ident. : M.M.D.  
Analysed : 04-02-97 13:44:08      Printed : 04-03-1997 14:08:39  
Sample Ident. : 12 PAINT CHIPS      Filename : RYNTA  
Sample Weight : 5.646      Calc.method: using 'Square to Linear fit'

Pk. (#)	Ret Time (Sec)	Area (fV/Sec)	Element % (%)	Area Ratio	Name
1	74	611	0.088	.539916E+03	Nitrogen
2	114	329943	11.289	.100000E+01	Carbon

Protein value = .5518133 (%)



Figure 13: Back Scatter Electron Image of Sample 59: Music Room Field

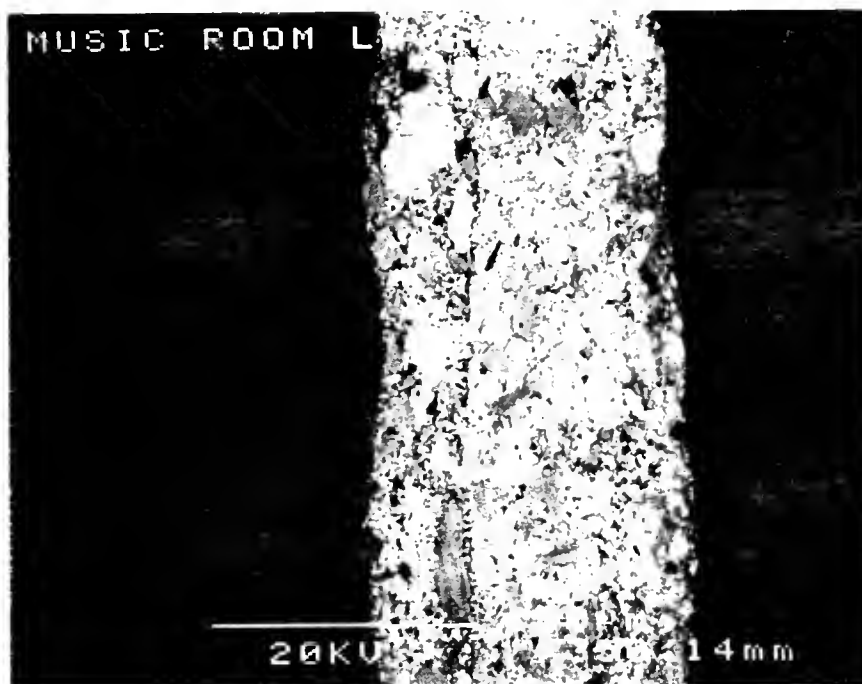


Figure 14: Secondary Electron Image of Sample 59: Music Room Field

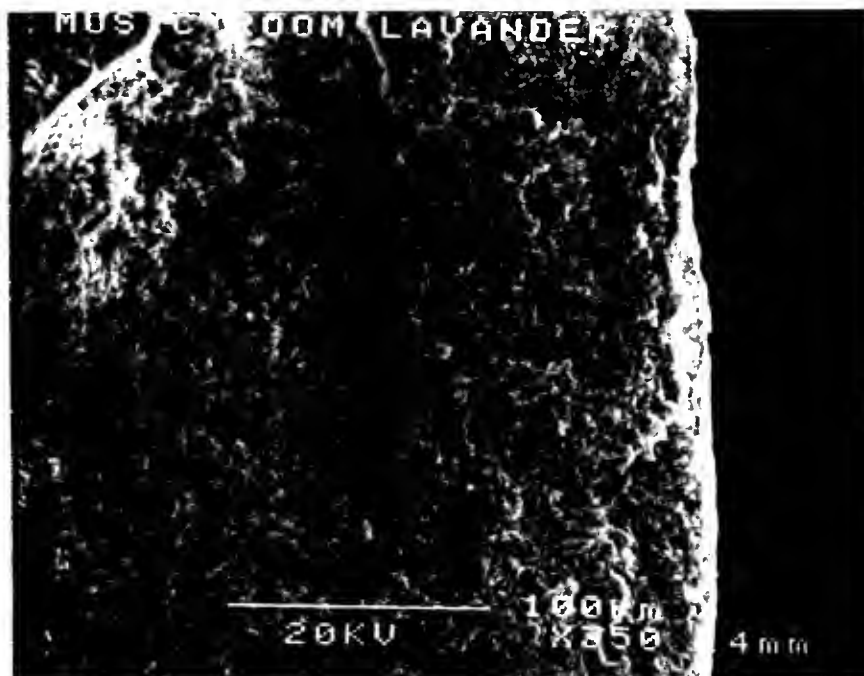




Figure 15: X-Ray Energy Dispersive Analysis of Sample 59: Music Room Field

Analysis of the field of the Music Room sample no. 59 indicated the presence of lithopone, iron oxide, ultramarine and whiting. This sample was carbon coated. The same sample was also gold coated.

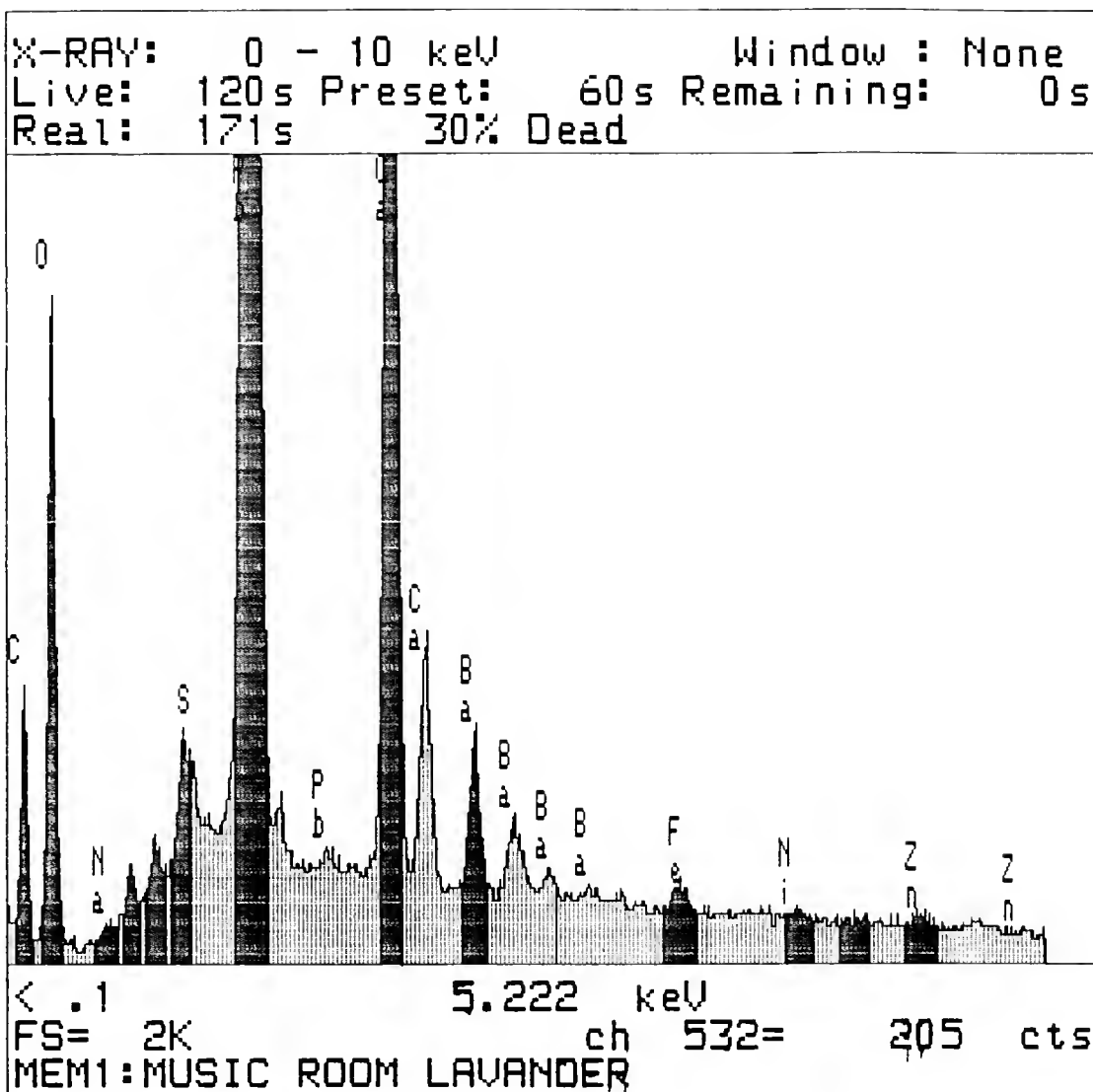
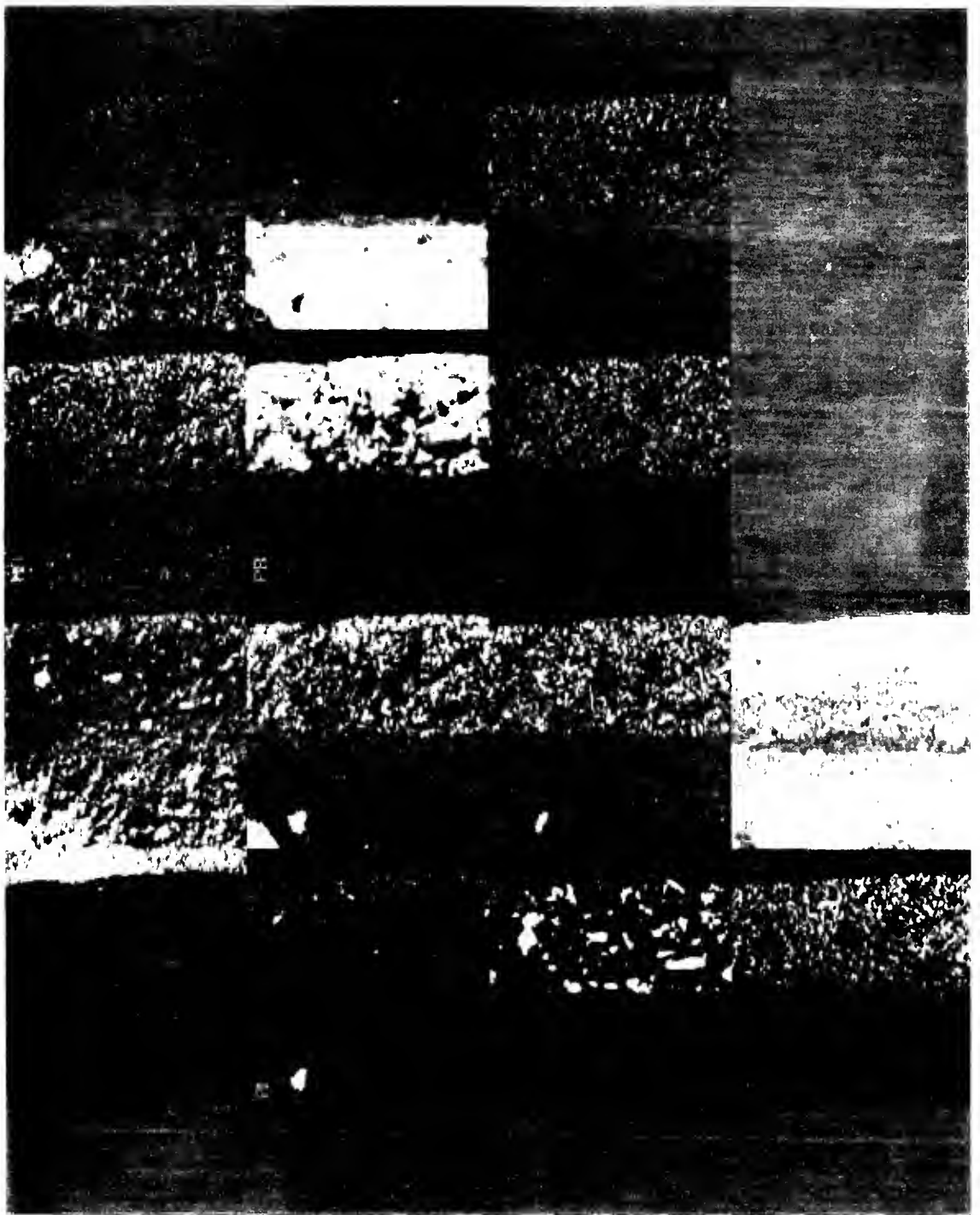






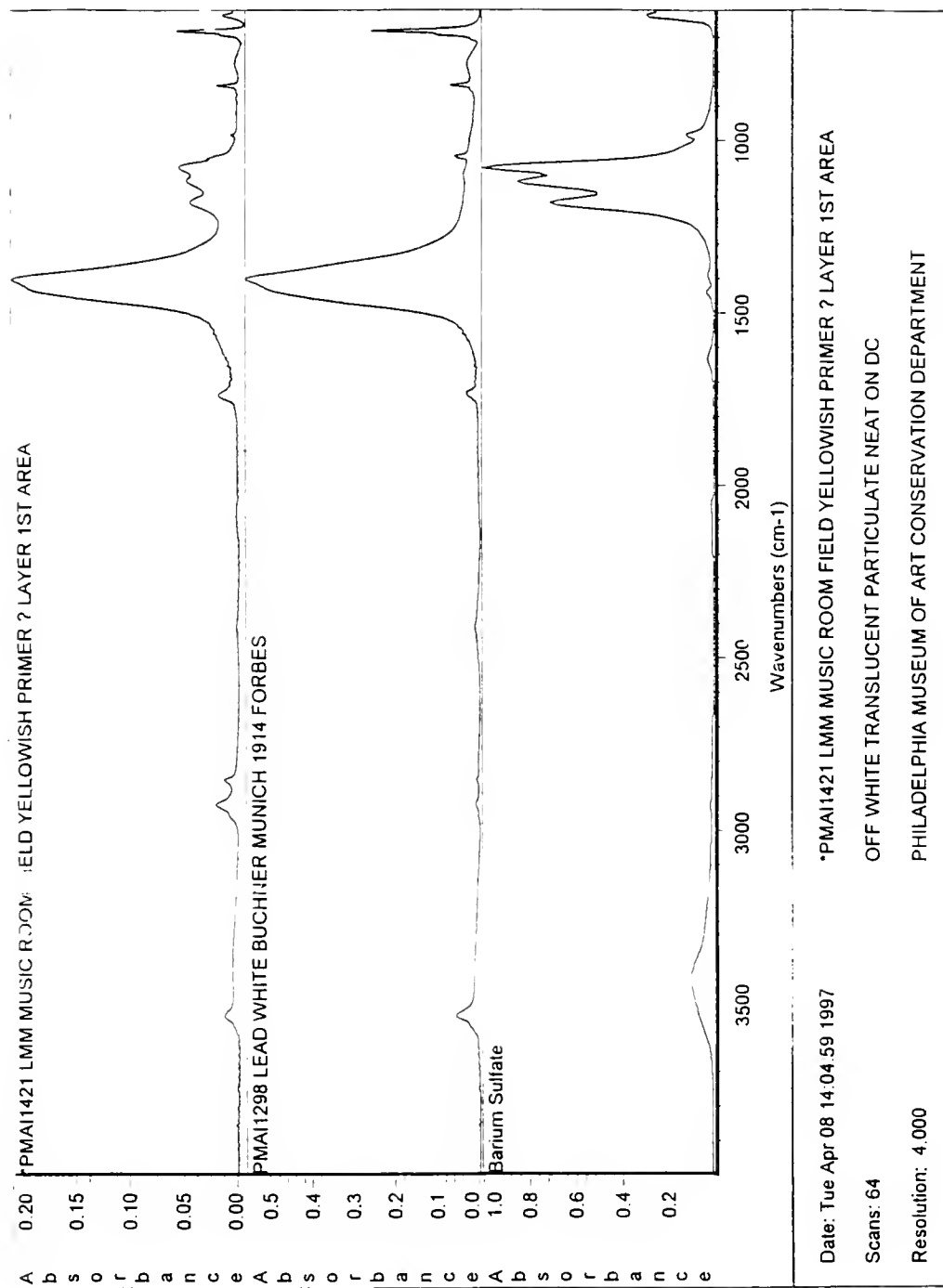
Figure 16: Digital X-Ray Mapping of Sample 59: Music Room Field





**Figure 17: FT-IR Insolubilizer of Paint. Sample 43: Music Room Field**

Indicating the use of lead white and baryte. It is also suggestive of lithopone if SEM result is interpreted with the FT-IR.





### 2.3. Pigments

Pigments were identified with chemical spot tests, optical microscopy, FT-IR, and SEM. The list of pigments identified is added in the Appendix. Table 1 shows the analytical methods used to identify the pigments found in the Lockwood-Mathews Mansion. Some pigments are inconclusive.

Ultramarine was identified with FT-IR, SEM, polarised light and chemical analysis. See comparative photographs of a known Ultramarine dispersed sample and the unknown blue pigment from the Music Room. (Fig. 18 - 20)

**Table 1: Analysis of Pigments**

PIGMENT	TEST	SAMPLE NO.
<b>GOLD - gilding</b>		
Gold with impurities	EDAX	27, 38
Copper with Ag	EDAX	46
Aluminium with Mo and Cr	EDAX	55
<b>WHITE</b>		
Baryte $\text{BaSO}_4$	FT-IR, Chem Tests, EDAX	36, 43, 45, 46, 49, 58, 9, 18, 33, 34
Lithopone $\text{ZnS BaSO}_4$	FT-IR, EDAX	42, 45, 46, 19, 21, 38
Whiting $\text{CaCO}_3$	EDAX, Chem Tests	46, 49, 1, 5, 21, 27
Gypsum $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$	EDAX, Chem Tests	59, 18, 33, 55
Lead White $2\text{PbCO}_3 \cdot \text{Pb(OH)}_2$	FT-IR, Chem Tests, EDAX, Optic	43, 45, 49, 59, 1, 9, 19, 26, 30, 38, 62
Lead Sulfate Basic $\text{PbSO}_4 \cdot \text{PbO}$	EDAX, Chem Tests	42, 45, 46, 58, 62, 18, 26
Zinc Oxide $\text{ZnO}$	EDAX, Chem Tests	21, 18, 26, 38, 39, 45
		43, 49
Titanium white	EDAX, Chem Tests	21, 27, 38



**Table 1: Analysis of Pigments cont.**

PIGMENT	TEST	SAMPLE NO.
<b>YELLOW</b>		
Yellow Ochre $\text{Fe}_2\text{O}_3 \cdot n\text{H}_2\text{O}$	EDAX, Chem Tests	46, 21, 30A
Barium Yellow $\text{BaCrO}_4$	EDAX, Chem Tests	21, 53
Chrome Yellow $\text{PbCrO}_4$	EDAX	21
Zinc Yellow $\text{ZnCrO}_4$	EDAX	21
<b>RED</b>		
Cuprous Oxide	EDAX	7
Red Ochre $\text{Fe}_2\text{O}_3$	EDAX, Chem Tests, Optical M.	42, 49, 33
Vermillion $\text{HgS}$	EDAX, Chem Tests, Optical M.	58, 18, 38, 29
Alizarin Crimson?	EDAX, Optical M.	9, 11, 21, 7, 29, 19
<b>GREEN</b>		
Green Earth $\text{KMg}(\text{Fe,Al})(\text{SiO}_3)_2 \cdot 3\text{H}_2\text{O}$	EDAX, Chem Tests	42, 32, 34
Verdigris	EDAX, Optical M.	3, 26
Zinc Green	EDAX	19, 34
Chromium Oxide $\text{Cr}_2\text{O}_3$	EDAX	21
Organic Green	EDAX	21
<b>BLUE</b>		
Phthalocyanine $\text{C}_{32}\text{H}_{16}\text{CuN}_8\text{Cl}_4$	EDAX	21
Ultramarine $\text{Na}_6(\text{Al}_6(\text{Si}_4)_6) \cdot \text{Na}_2\text{S}_2$	FT-IR, Chem Tests, EDAX, Optic	42, 45, 1, 21, 28, 41
Prussian Blue $\text{Fe}_4(\text{Fe}(\text{CN})_6)_3$	EDAX, Chem Tests, Optical M.	28
Lead blue?	EDAX	1
<b>BLACK</b>		
Carbon Black C	EDAX	5, 55
Lead black	EDAX	5, 37
Iron Oxides	EDAX	37





**Figure 18 and 19: Known Ultramarine Dispersed Sample Compared To The Unknown Blue Pigment Particles From The Music Room Field.**

**Sample 45**

**Sample Location:** Music Room field, blue line.

**Type of Film:** 200 ASA Royal Gold, Film 7 Negative 4 and 5

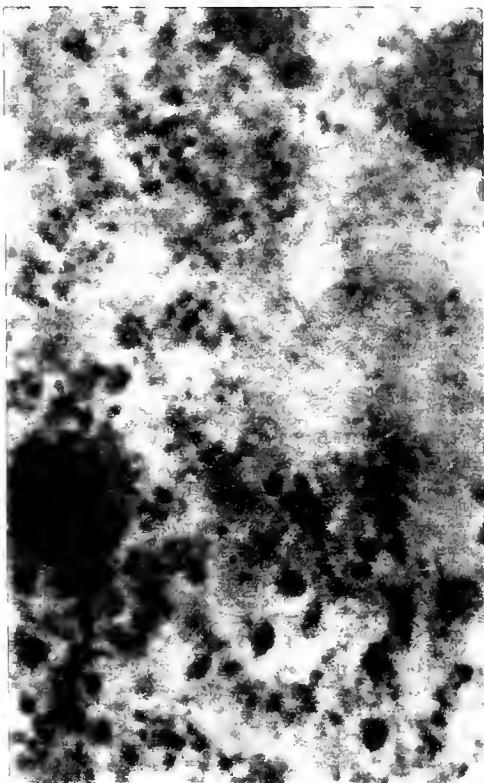
**Magnification:** 125X

**Camera:** Nikon

**Polarized Light**

Both samples were dispersed in melt mount and seemed to have the same refractive index.

**Known**



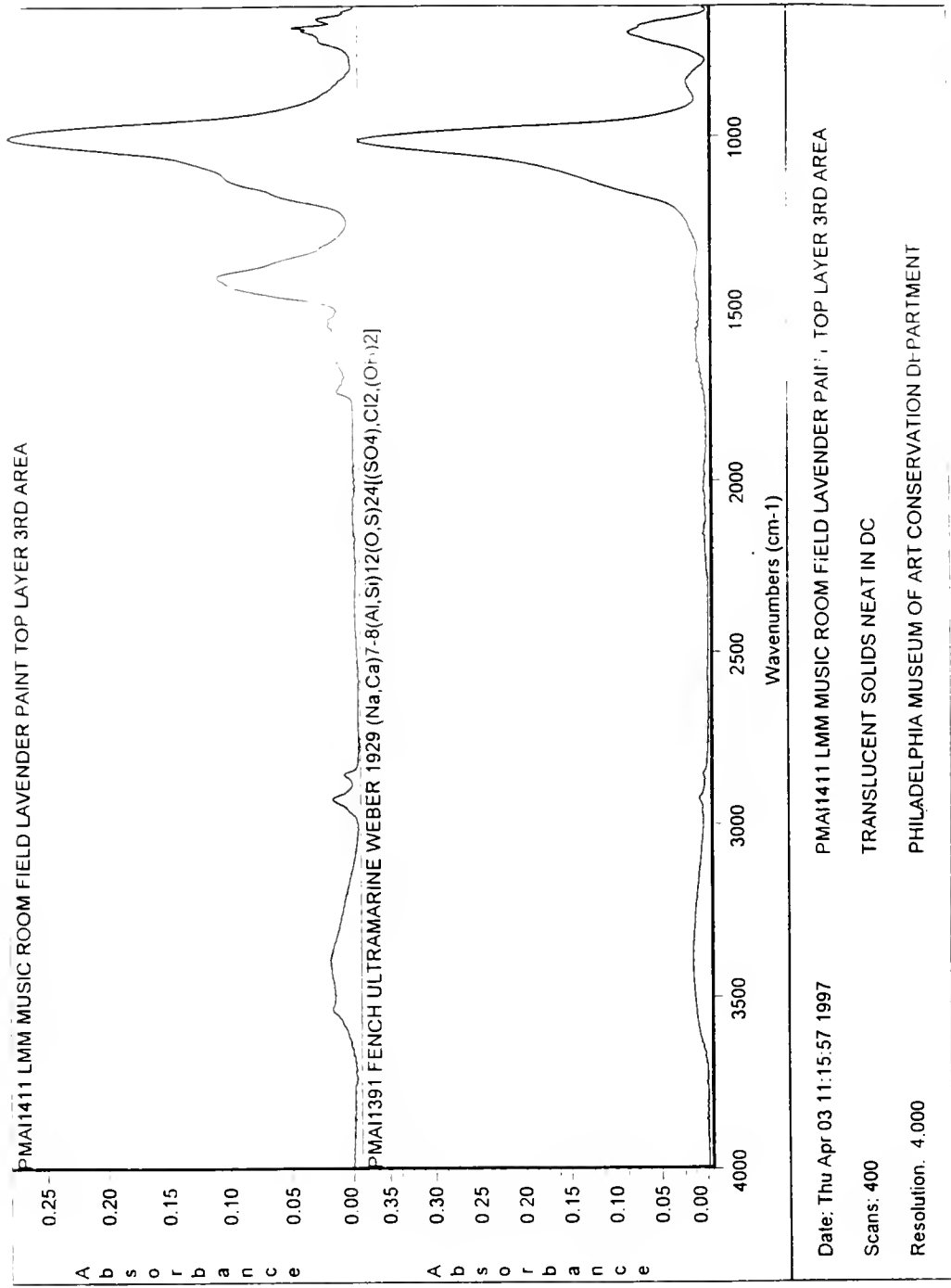
**Unknown**





Figure 20: FT-IR Indicates The Suggestive Presence Of Ultramarine

Top graph is sample 43 from the Music Room and the bottom graph is the standard for ultramarine.





## **2.4. Ready-Mixed versus Hand-Mixed Paints**

Many questions arise concerning the paint in the Lockwood-Mathews Mansion. Was this paint manufactured in the United States? Were these paints Hand-Mixed or Ready-Mixed?

In comparison to paint recipes found in the literature from 1868, the paints in the Mansion seem to be of a later date and better quality. However, some literature mentions that England and Germany were more advanced with casein paints than the United States during this time. The paints used on the Mathews-Lockwood mansion might have been imported as the Herter Brothers originated from Germany; and at this particular time, one of the brothers went back to Germany. Moreover, Germany was already producing lithopone. However, these are only speculations which may never be fully answered.

As to whether the paints were ready-mixed or hand-mixed was determined by optical microscopy. Paint on large areas such as the field of a design which has small, regular-shaped, well-dispersed pigments, indicates that ready-mixed paints were used. However, paint on small detail areas as found on the ceilings have irregular shaped pigment particles which indicate that hand-mixed paint was also used at the mansion. The paints used in the Mathews-Lockwood Mansion are intriguing as they represent a transitional phase in the paint-making industry. (Fig. 21 and 22)



**Figure 21: Hand-Mixed Paint of Sample 20B: Mrs. Lockwood's Room**

Paint from areas where the design is intricate on the ceilings and walls seems to be painted with hand-mixed paint.

**Sample 20B**

**Sample Location:** Mrs. Lockwood's Room-ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 7 Negative 26

**Magnification:** 125X

**Reflected Light**

**Camera:** Nikon



Uneven  
particles  
distribution  
and shape,  
looks like  
hand made  
paint.

**Figure 22: Ready-Mixed Paint of Sample 5A: Mrs. Lockwood's Room**

Paint from large areas such as the field seems to be painted with Ready-Mixed paint.

**Sample 5A**

**Sample Location:** Mrs. Lockwood's Room - wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 7 Negative 27

**Magnification:** 125X

**Reflected Light**

**Camera:** Nikon



Even  
distribution  
of particles  
and shape.





## **2.5. Designers**

Of the seventeen rooms studied in the Mathews-Lockwood Mansion, only five rooms have known designers

List of the seventeen rooms:

1. Music Room
2. Library
3. Rotunda
4. Mrs. Lockwood's Room
5. Mrs. Lockwood's Washroom
6. Bathroom of Mrs. Lockwood's Room
7. Connecting Room to Mr. Lockwood's Room
8. Mr. Lockwood's Room
9. Mr. Lockwood's Bathroom
10. Oratory
11. Storage Room part of Mr. Lockwood's Room
12. Moorish Room
13. Mirror Rooms or Twin Rooms
14. Italian Suite
15. Passage around Rotunda
16. Secondary Staircase
17. Wash Room



Samples from these rooms were studied in order to see if the designers of the other rooms could be identified. However, definite conclusions were difficult to make after using the following samples, as knowns.

- Leon Marcotte: Library, Samples 55 and 66
- Herter Brothers: Music Room and Italian Suite, Samples 42 - 50
- George Platt: Moorish Room, Samples 37, 38, and 61
- Hutchingson & Son: Twin Rooms, Sample 62 (Fig. 4 and 6)

The conclusion that could be made after comparing the knowns and unknowns was that the Hallway around the rotunda on the second floor and Mrs. Lockwood's room were done by the Herter Brothers. (Fig. 23 and 24). The same type of paint technique and pigments were used in these two rooms as in the Music Room. However, differences in technique, material, and style exist even between the Music Room and the Italian suite. This indicates that the Herter Brothers were truly masters in their craft and could well have done other rooms.



**Figure 23: Photo Micrograph of Sample 1: Mrs. Lockwood's Room**

It seems that the technique and paint were done by the same designer, in this case the Herter Brothers. Note the primer or first paint layer. This is found in several cross sections. This layer is very similar to the next layer. (Appendix, FT-IR of Music Room.)

**Sample 1**

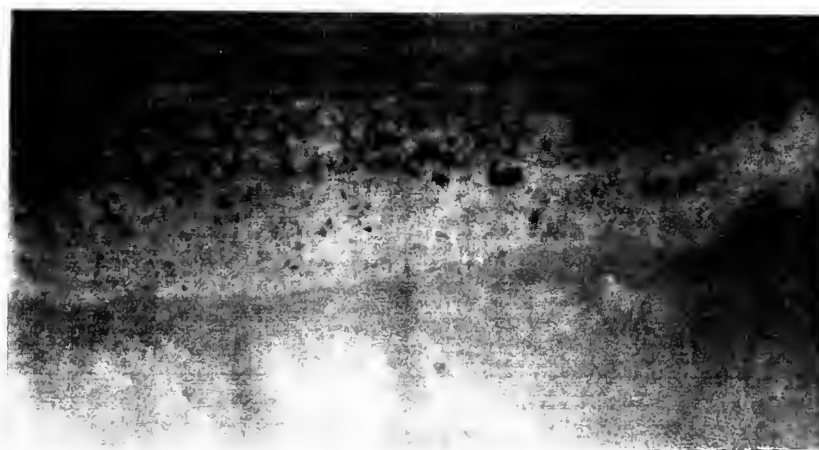
**Sample Location:** Mrs. Lockwood's Room

**Type of Film:** 200 ASA Kodak Royal Gold, Film 5 Negative 8

**Magnification:** 225X

**Reflected Light**

**Camera:** Nikon



Ultramarine,  
part of small  
stencil design

Paint Layer  
of the field

Primer,  
similar type  
of paint as  
the final  
layer.

**Figure 24: Photo Micrograph of Sample 28: Hallway**

**Sample 28**

**Sample Location:** Hallway

**Type of Film:** 200 ASA Kodak Royal Gold, Film 5 Negative 21

**Magnification:** X220

**Reflected Light**

**Camera:** Nikon



Ultramarine,  
part of small  
stencil design

Paint Layer  
of the field

Primer,  
similar type  
of paint as  
the final  
layer.



## **2.6. Diachronic (through-time) Interpretation of Campaigns**

The oldest paint sample that was found is a distemper type paint in the wash room: Sample 64., pigment: yellow ochre. It is reasonable to conclude that a preliminary paint was applied because the Lockwood's moved into the house before the interior finishes were completed. Another indication of an early intermediate campaign is a two layer preliminary coat with a scraped-off white distemper type paint found in Mrs. Lockwood's Room. This color is particularly evident on the first floor, and Mary Findlay mentions that she has found this color frequently.

The choice of using casein might be based on the Lockwood's early move-in before completion. The smell and long drying time necessary for oils would explain the use of casein rather than oil-based paints. It is not possible to determine the chronological sequence for decorating the house, but one can safely assume that the first floor would receive priority. This would explain why the Moorish room was never completed due to Lockwood's losses in 1869. It is known that the Mathews did not redesign any of the interiors except the rotunda which was repainted after 1878. The City of Norwalk added several paint campaigns which are easily recognizable because they are emulsion paints.




## **2.7. Interpretation of the Paint in the Different Rooms**

To illustrate the major colors found in the rooms, paint chips were prepared with casein as a binder using almost all the identified pigments. These main colors found in the rooms were compared to the Munsell colors. Pigments such as lead and vermillion were not used. When an pigment was added that was not identified, the pigment's name is in italics. (Table 2-12)





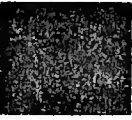





**Table 2: Music Room**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Gold Leaf alloy Zn, Sn, Cu	46	gold				1868
Vermillion Barytes Lithopone Whiting	58	red rosette	7.5R 4/8		casein	1868
Iron Oxide Carbon Black Barytes Lithopone	50	light brown red rosette	7.5YR 5/6		casein	1868
Probably same as brown, more whites	50	light brown	not available		casein	1868
Iron Oxide Lead Oxide Ultramarine No Lithopone Barytes Whiting Alizarin Crimson Sodium ?	61, 43, 63	field grey lavender	8.13R 8.19/1.127		casein	1868
?	49, 58	beige border	9.31Y 8.12/0.78		casein	1868
Ultramarine Yellow Ochre Lithopone Barytes Whiting	44	green leaves	7.5Y 5/2		casein	1868
Ultramarine Azurite	45	blue line	5PB 7/2		casein	1868

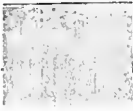
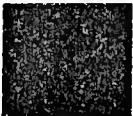


**Table 3: Mrs. Lockwood's Room**


Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Green Earth Carbon Black CI?	5	black	N2.25/		casein	1868
Same as above more Whiting	5	grey beige	10R 8/1		casein	1868
Ultramarine Red or Alizarin Crimson? Lithapone	18	3D/Red	10RP 6/4 10RP 6/2 10RP 4/4		casein	1868
Alizarin Crimson Lithapone Iron Oxide	19, 21	brown- purple (ceiling)	5YR 6/4		casein	1868
Titanium White Ultramarine Green Earth Verdigris	21	green touch-up	7.5Y 5/2		?	c. 1920s
Ultramarine	1	blue below cornice	5PB 6/4		casein	1868




**Table 4: Bathroom of Mrs. Lockwood's Room**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Whiting	27 Bath Surface	beige	2.5Y 8.5/2		casein	1868
Gold with Alloys Cr, Zn	27 Bath Surface	gold				1868
Lithopone Zinc Oxide Yellow Ochre Lead Oxide Yellow	8, recent layer	dark grey	N5.75/		emulsion paint?	c. 1900s

**Table 5: Bathroom of Mr. Lockwood's Room**


Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Zinc Oxide No Barytes No Lithopone Green Earth Cl, Ni?	39	green field	10GY 6/2		casein	1868

**Table 6: Passage**


Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Ultramarine <i>Prussian Blue</i> <i>Iron Oxides</i> No Silica	28	blue	5PB 5/4		casein	1868




**Table 7: Staircase**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Prussian Blue <i>Iron Oxides</i> <i>Alizarin Crimson</i>	41	blue	5PB 6/4		casein	c. 1876


**Table 8: Library**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Carbon Black Vermillion Burnt Umber Yellow Ochre	55	brown/ graining	7.5YR 3/2		?	1868
Gold not tested	56	gold			glazing	1868

**Table 9: Mirror or Twin Rooms**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Lead Oxide Alizarin Crimson? Zn, Cl?	62	field	N6.0/		casein?	1868


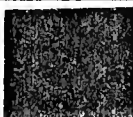



**Table 10: Rotunda**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Aluminum Leaf with Alloys Mo and Cr	53	gold			glazing	1876
Lithopone Alizarin Crimson Carbon Black Yellow Ochre	51	red	2.5R 6/4		casein	1868





**Table 11: Moorish Room**

Pigments	Sample No.	Color	Munsell Color	Paint Chip	Binder	Period
Black Lead Iron Oxide Carbon Black Vermillion? Dolomite?	37, 38	black	N2.25/		casein	1868
Same as above but more whiting	37, 61	grey field	7.5PB 6/2		casein	1868
Gold with copper, lead, Ba and Cu?	37	gold				1868
Vermillion mixed with Alizarin Crimson	38	orange	7.5R 4/8		casein	1868
Same as above but less sulphur, no Hg	38	beige			casein	1868



### 3. Substrate

The substrate of the paint consists of two parts, the scratch coat and the finish coat. The thickness of the scratch coat varies, but the average thickness is 18 mm.

The finish coat varies in thickness between 2 mm - 10 mm. The finish coat is gypsum. The finish coat for the ceilings is different than the finish coat for the walls. The latter has small quartz particles whereas the other has none.

The examination of the scratch coat revealed that the binder of this plaster is calcium carbonate. XRD verified this analysis. The mortar analysis showed that the plaster consists of 16.53% binder, 79.78% sand, and 3.7% fines.

In the scratch coat, fiber for strengthening the plaster was found. It was tested chemically to determine whether it was animal or vegetable fiber. This was done by placing the fiber on a spot plate adding water and one drop of iodine-azide. If after a minute, the fibres were surrounded by gas bubbles, the fiber is of animal origin. The hair was cleaned in a ultra sound bath and studied with SEM. It contains a large amount of magnesium. The distance between the external margins of the cuticular scales of the hair are intermediate. The type of margin of the cuticular scales are rippled. The pattern of the cuticular scales are double chevron.<sup>100 101</sup> The diameter of the hair is 50  $\mu\text{m}$ . The length varies between 5-20 mm. It has been tentatively identified as horse hair.

By comparison of the thin sections, it is concluded that all the plasters in the Lockwood-Mathews Mansion are the same. (Fig.24 - 38)

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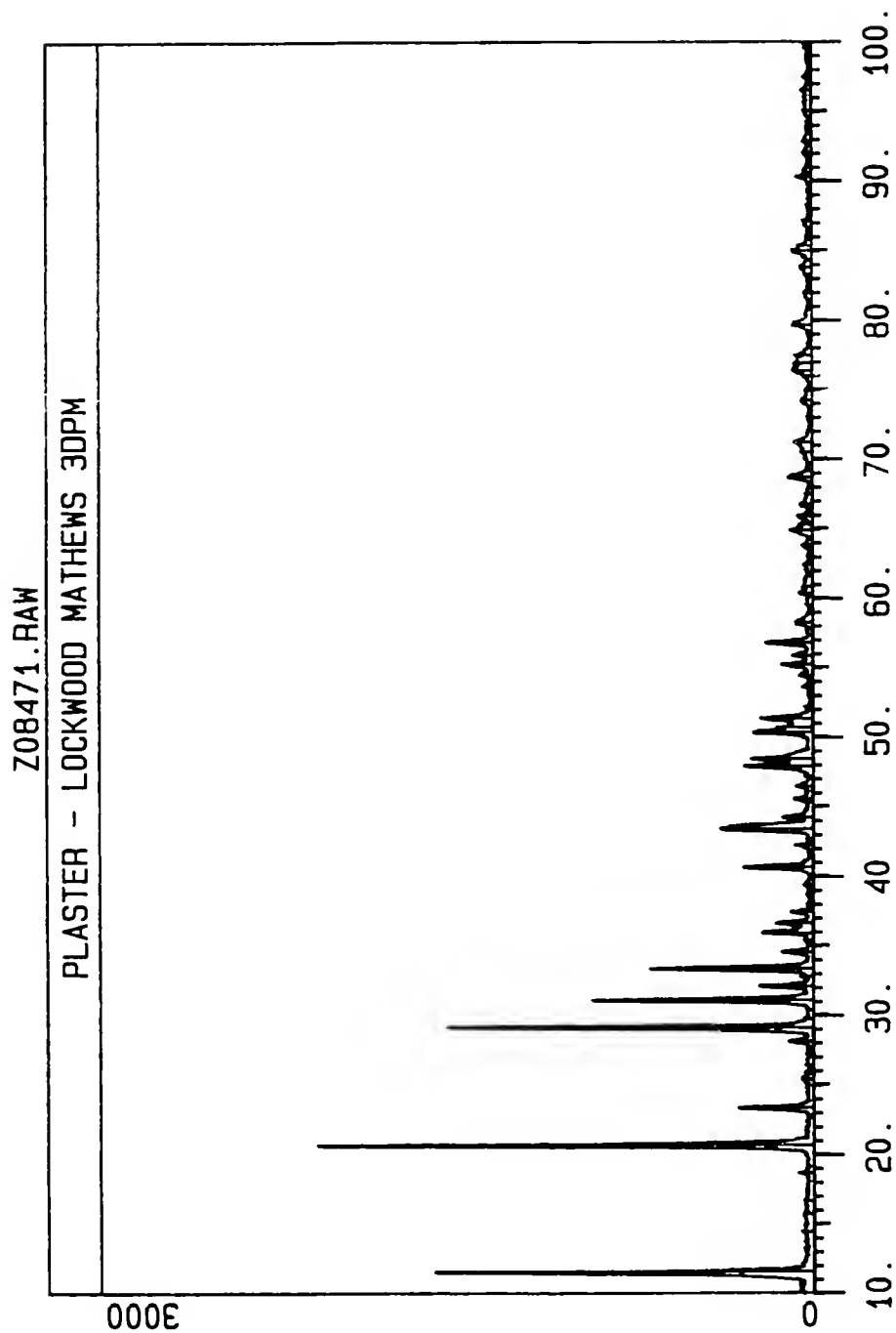
<sup>100</sup>Terminology used by Wildman in 1954 from *the Study of identification Characteristic of Mammal Hair*. Wyoming Game and Fish Laboratory, 1963, p.16.

<sup>101</sup>McCrone, Walter. *The Particle Atlas ed. 2. An Encyclopaedia of Techniques for Small Particles Identification*. Vol V., Ann Arbor Science Publ. Inc., MI, 1979, p. 1383.



Figure 25: XRD of Sample 7: Mrs. Lockwood's Room

Indicates gypsum and calcite in the finish coat.

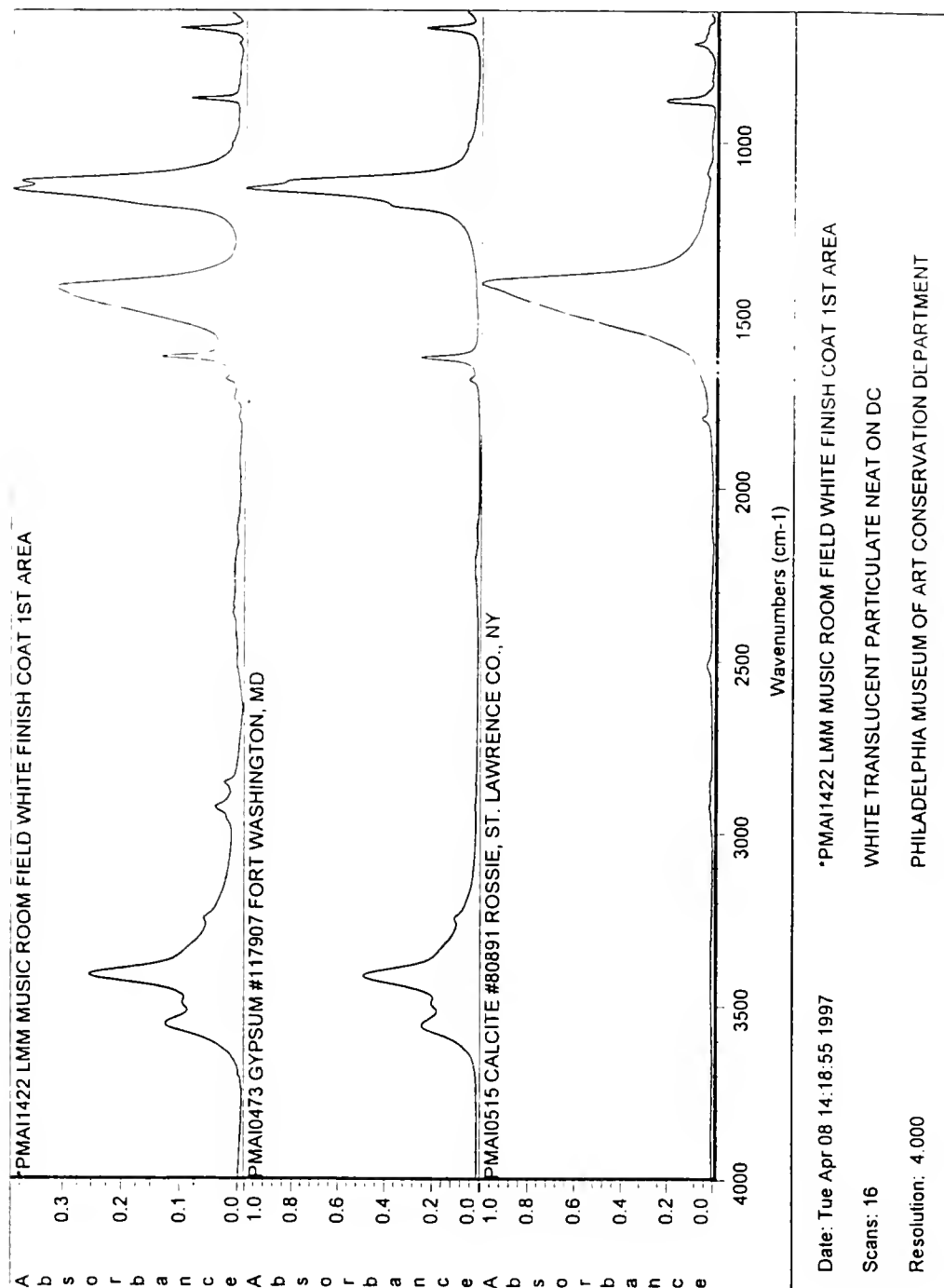




**Figure 26: FT-IR of Sample 43: Music Room**

Indicates gypsum and calcite in the finish coat. Top graph is Sample 43. Music Room.

The second and third is standards for gypsum and calcite respectively.







**Figure 27: Thin Section of Plaster - Sample 8: Mrs. Lockwood's Room**

**Sample 8**

**Sample Location:** Mrs. Lockwood's Room

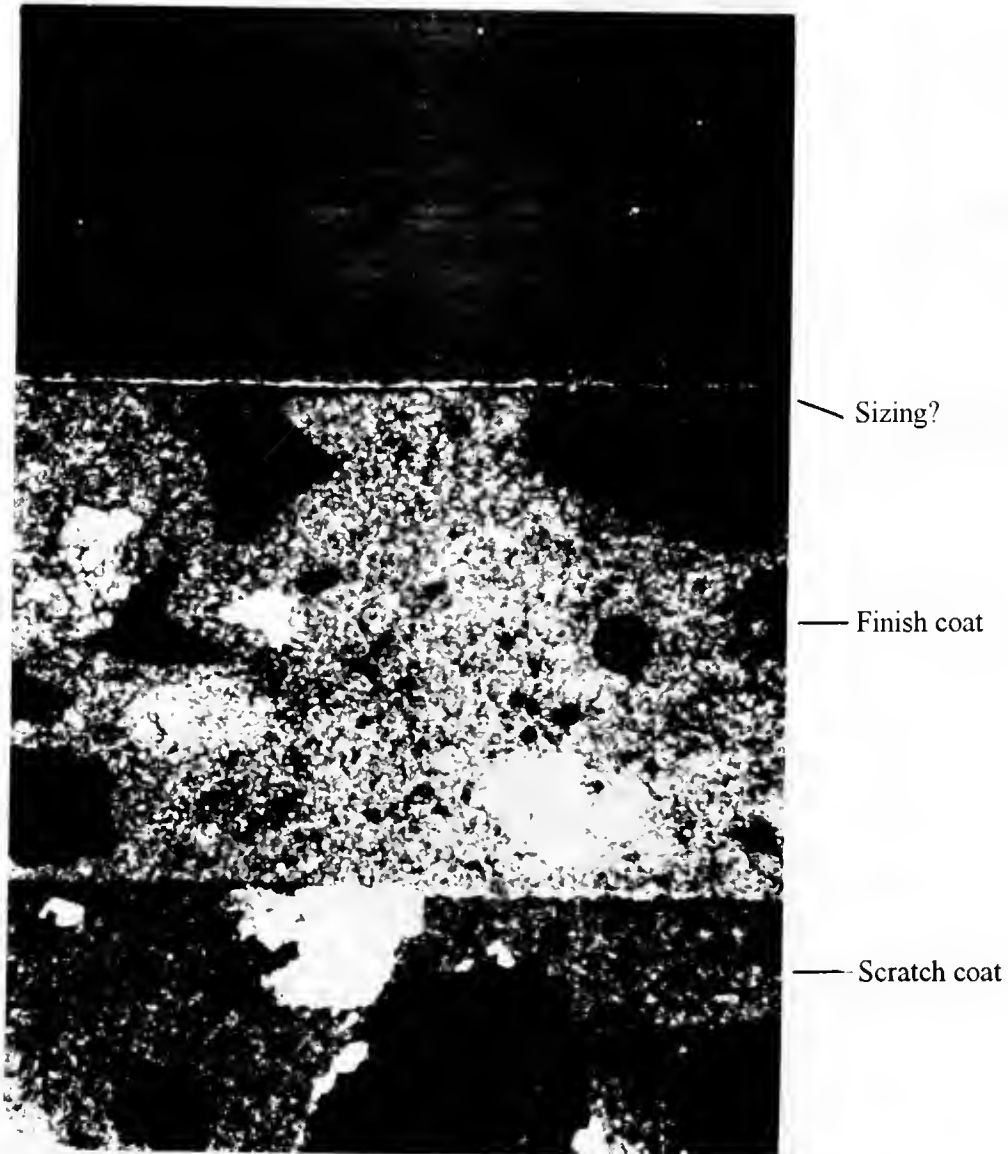
**Type of Film:** 200 ASA Kodak Royal Gold, Film 7 Negative 3

**Magnification:** 125X

**Polarized Light**

**Camera:** Nikon

**Crossed Nicols**





**Figure 28: Photo Micrograph of Sample 8: Bathroom of Mrs. Lockwood's Room**  
Cross Section of Scratch Coat.

**Sample 8**

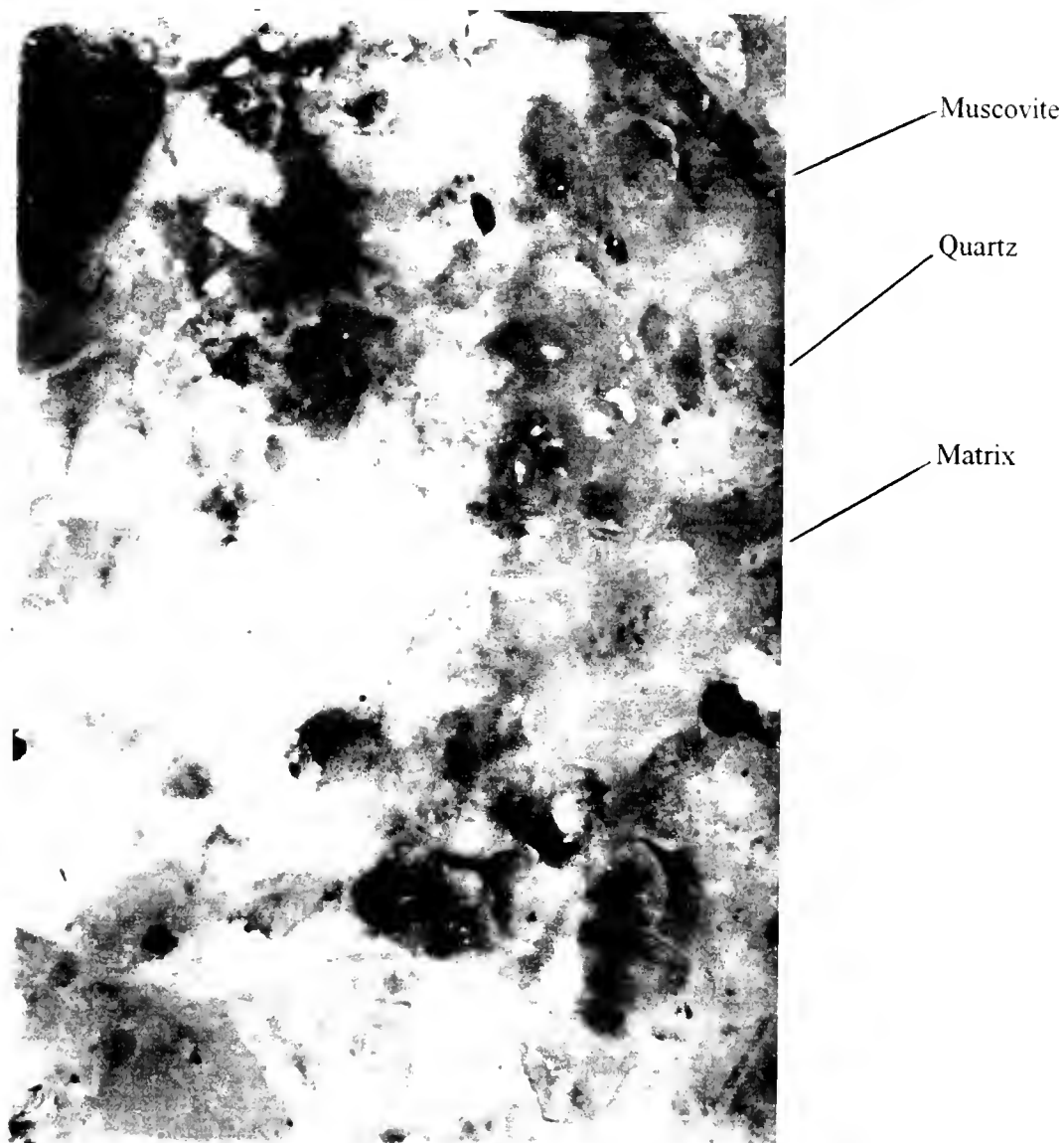
**Sample Location:** Bathroom of Mrs. Lockwood's Room - wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 7 Negative 18

**Magnification:** 125X

**Reflected Light**

**Camera:** Nikon





**Figure 29: Thin Section of Scratch Coat - Sample 8: Mrs. Lockwood's Room**

**Sample 8**

**Sample Location:** Mrs. Lockwood's Room - wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 7 Negative 14

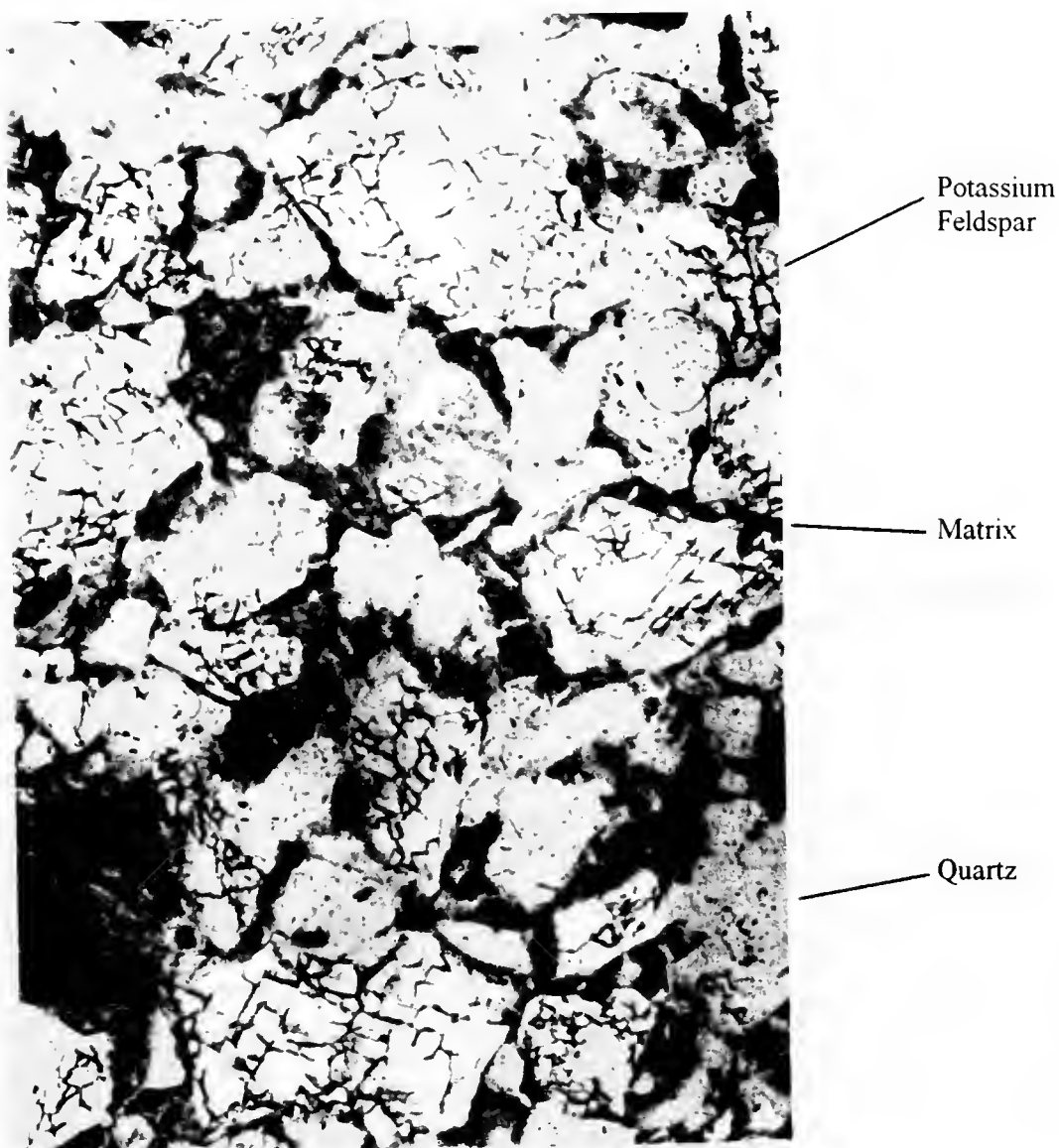
**Magnification:** 125X

**Polarized Light**

**Camera:** Nikon

**Parallel nicols**

Potassium Feldspar and Quartz are both fractured. The particles are not rounded; they appear to be crushed stone and not river sand.





**Figure 30: Thin Section of Scratch Coat - Sample 8: Mrs. Lockwood's Room**

**Sample 8**

**Sample Location:** Mrs Lockwood's Room - wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 7 Negative 15

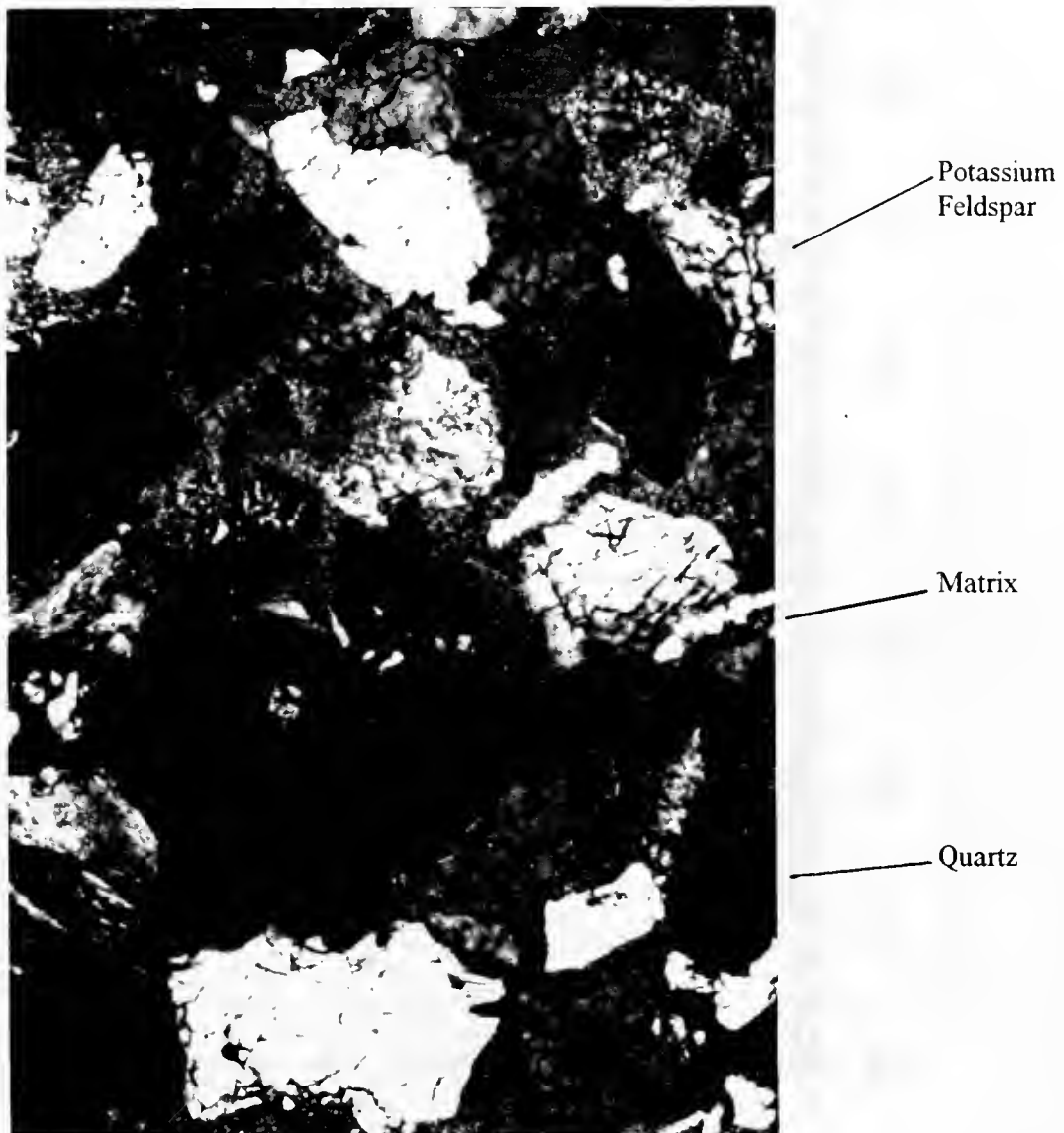
**Magnification:** 125X

**Polarized Light**

**Camera:** Nikon

**Crossed nicols**

Potassium Feldspar and Quartz are both fractured. The particles are not rounded; they appear to be crushed stone and not river sand.

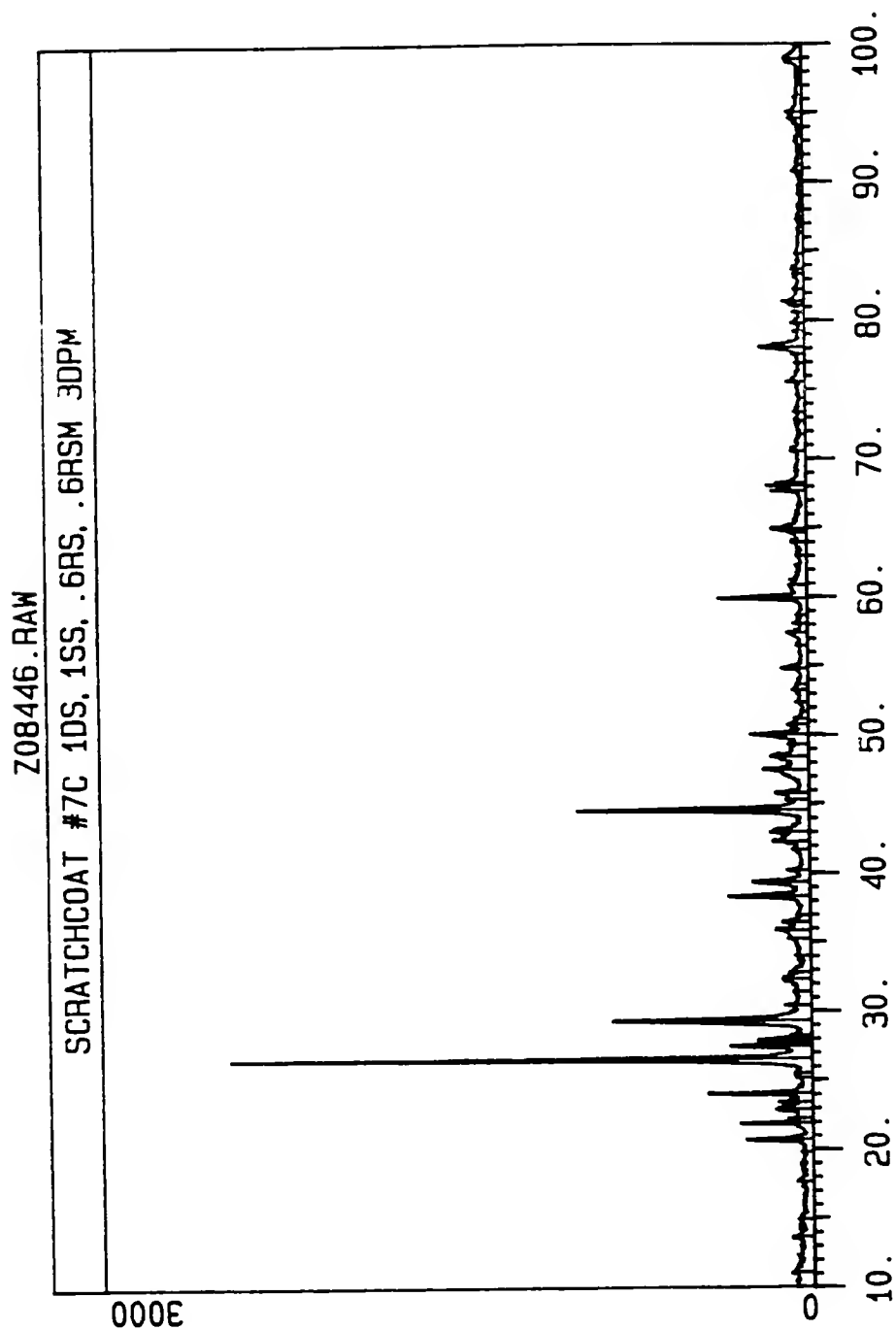






**Figure 31: XRD of Scratch Coat - Sample 7C: Bathroom of Mrs. Lockwood's Room**

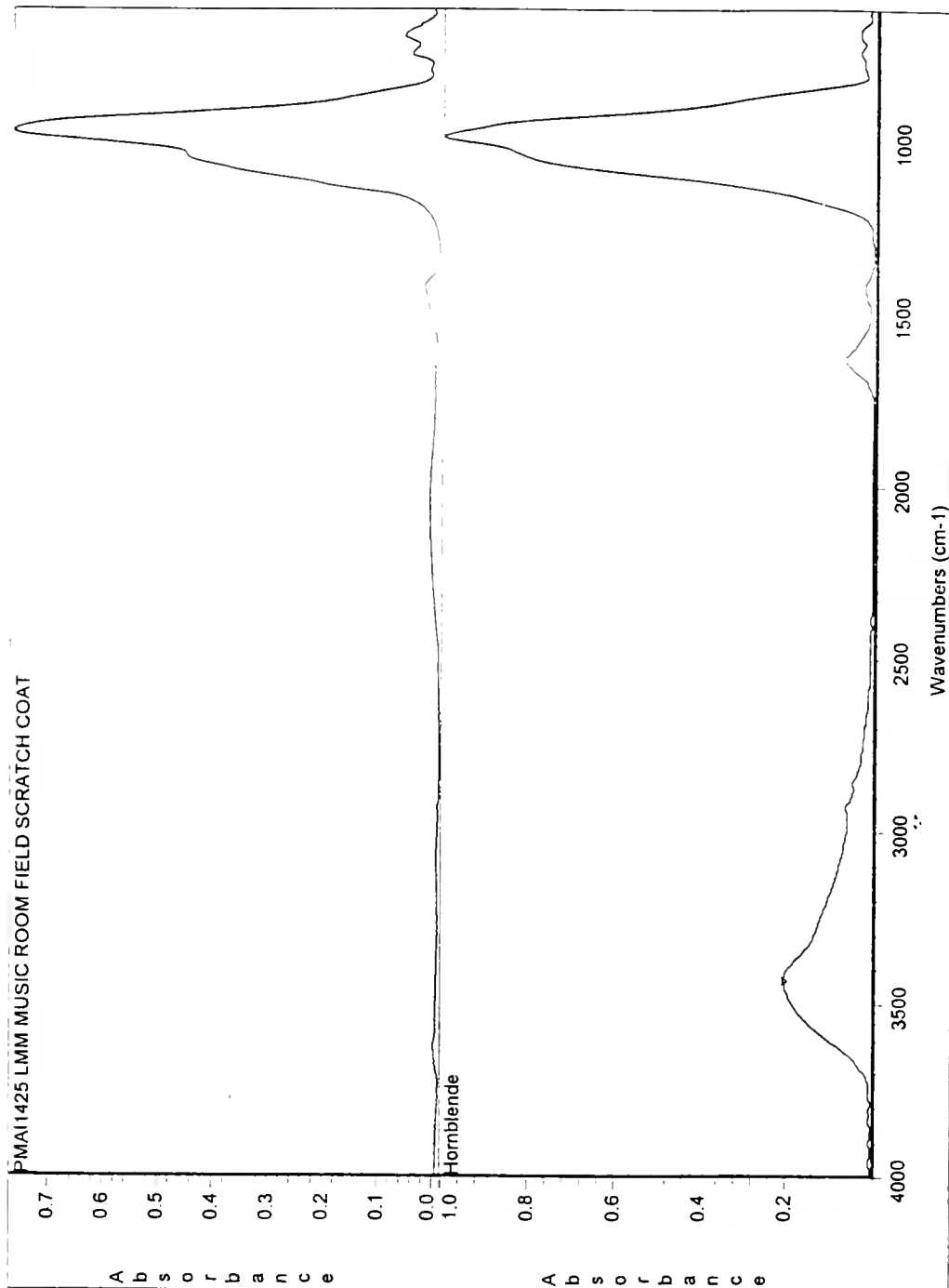
The Sample 7C was sifted. The pan was used for XRD. This indicates the use of calcium carbonate as binder for the plaster.





**Figure 32: FT-IR of Particles Found in the Scratch Coat - Sample 43: Music Room**

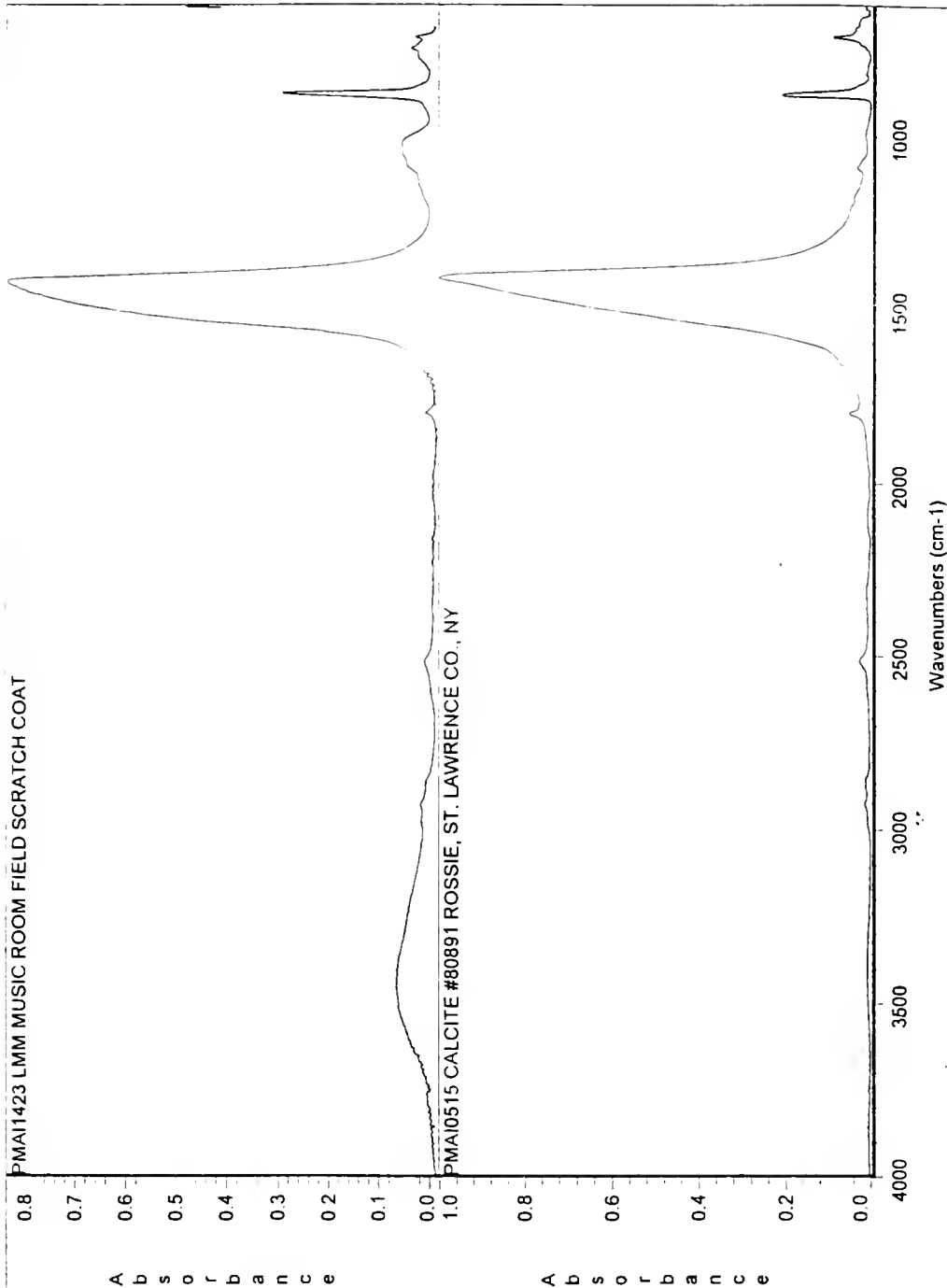
Comparing a grain of the scratch coat in Sample 43: Music Room in the top graph to a standard of Hornblende. It is suggestive of hornblende.





**Figure 33: FT-IR of Particles Found in the Scratch Coat - Sample 43: Music Room**

Comparing a grain of the scratch coat in Sample 43: Music Room in the top graph to a standard of calcite. It is found to be calcite.





**Figure 34: FT-IR of Particles Found in the Scratch Coat - Sample 43: Music Room**

Comparing a grain of the scratch coat in Sample 43 Music Room in the top graph to two standards of muscovite.

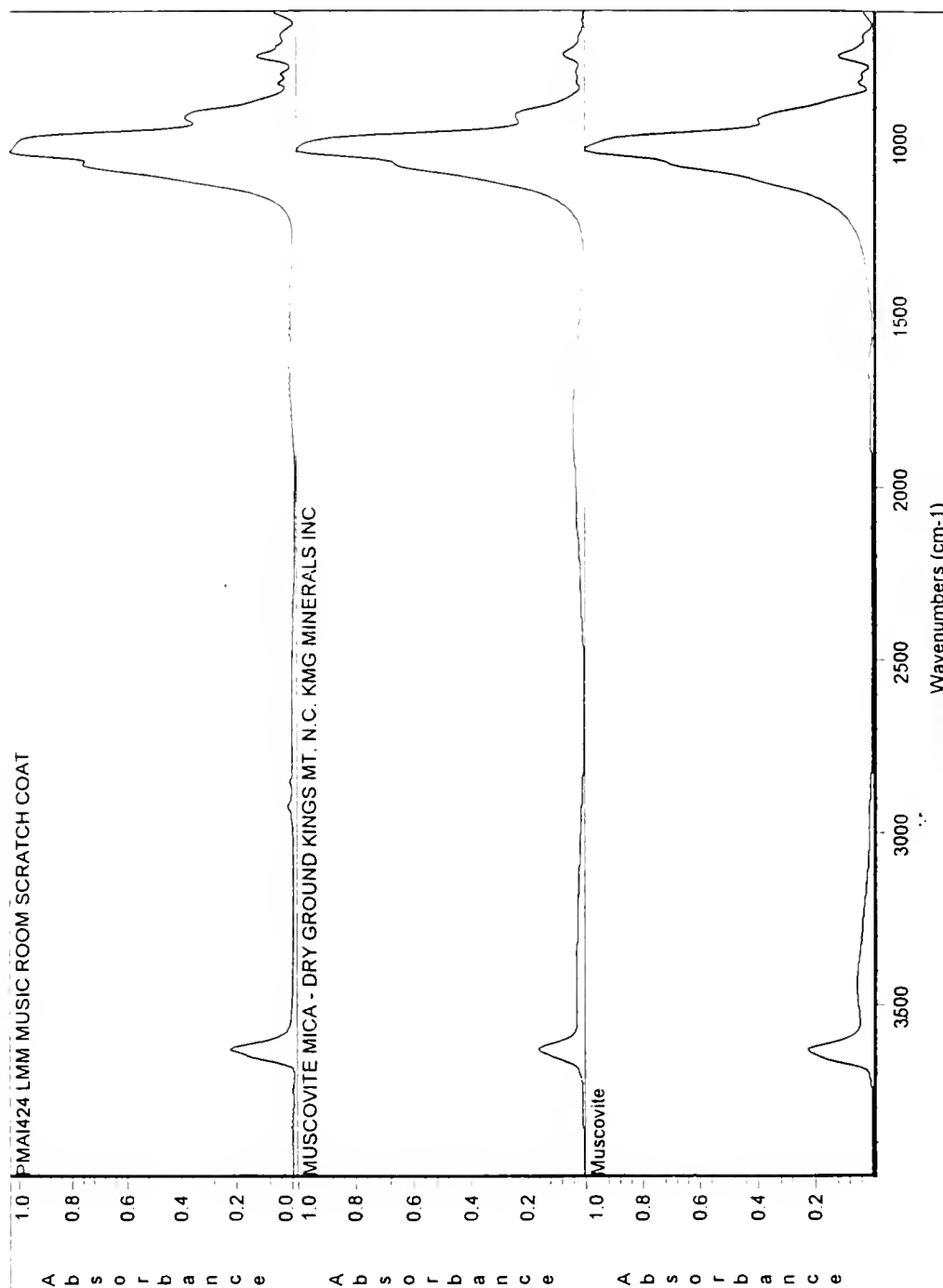


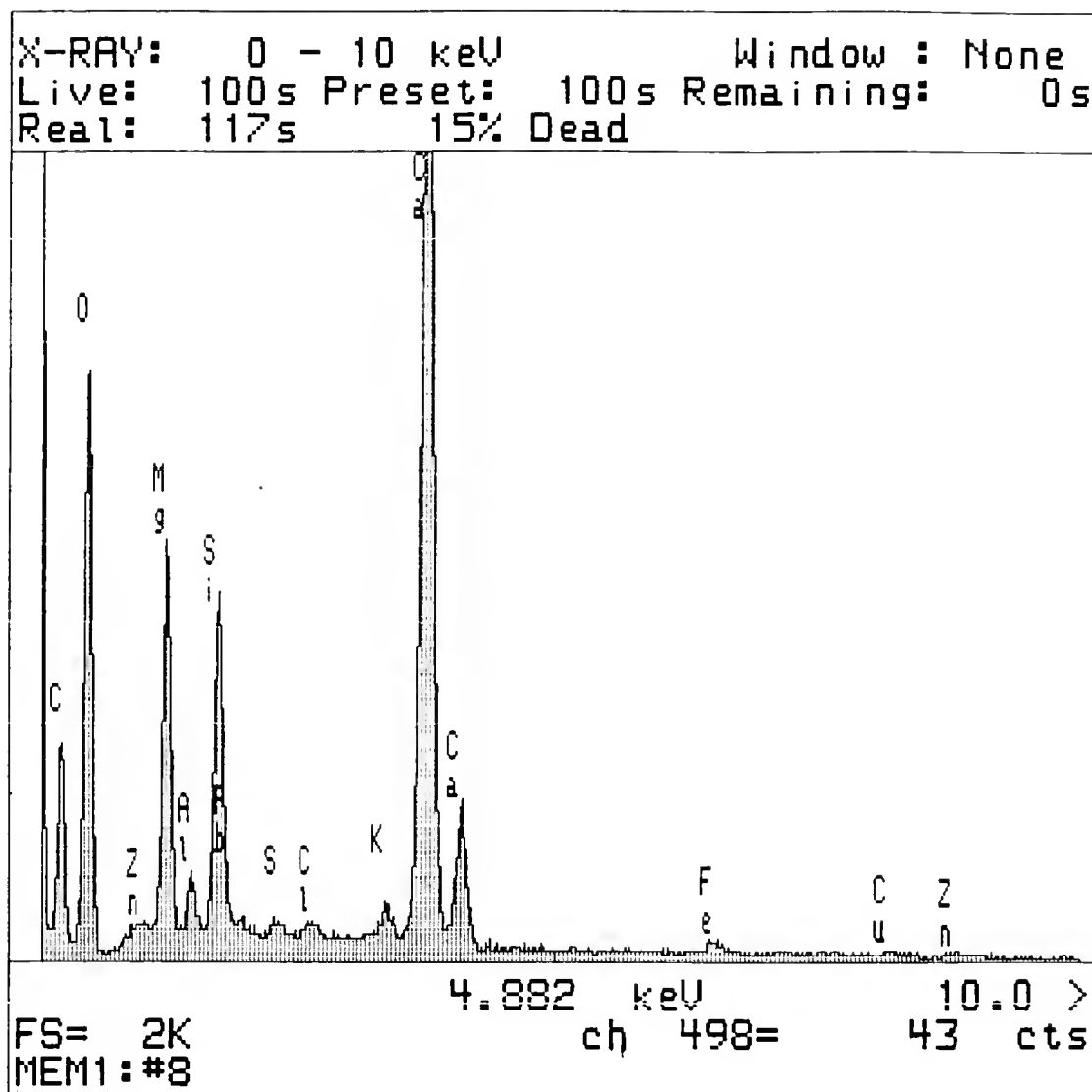




Figure 35: X-Ray Energy Dispersive Analysis of the Scratch Coat - Sample 8: Mrs.

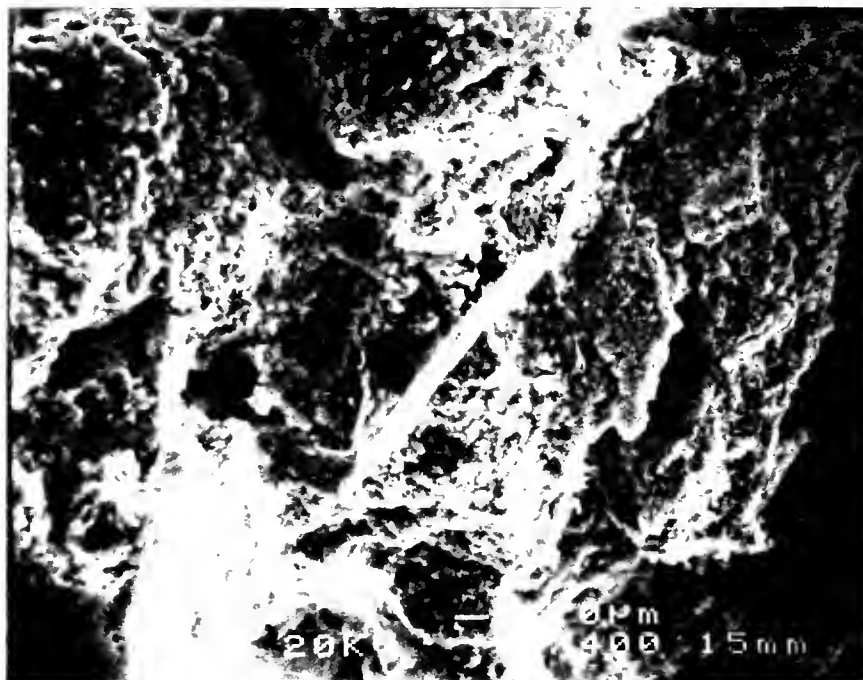
Lockwood's Room

Confirms the presence of calcium carbonate, quartz and potassium feldspar.

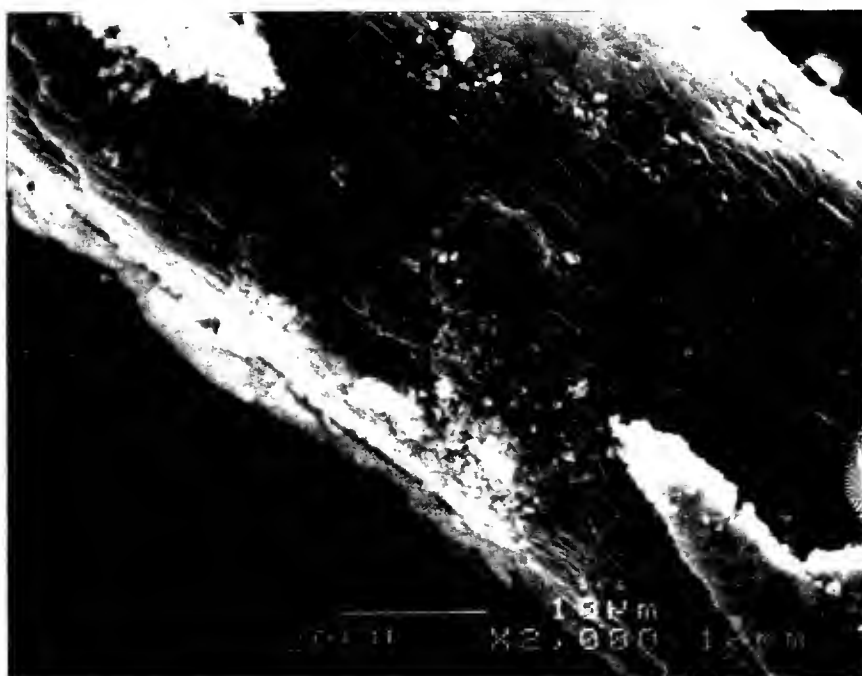




**Figure 36: Secondary Electron Image of the Scratch Coat - Sample 8: Bathroom of Mrs. Lockwood's Room**



**Figure 37: Secondary Electron Image of Fiber Found in Plaster - Sample 28: Hallway**





**Figure 38: Fiber Found in Plaster - Sample 28: Hallway**

**Sample 28**

**Sample Location: Hallway**

**Type of Film: 200 ASA Kodak Royal Gold, Film 7 Negative 1**

**Magnification: 125X**

**Polarized Light**

**Camera: Nikon**

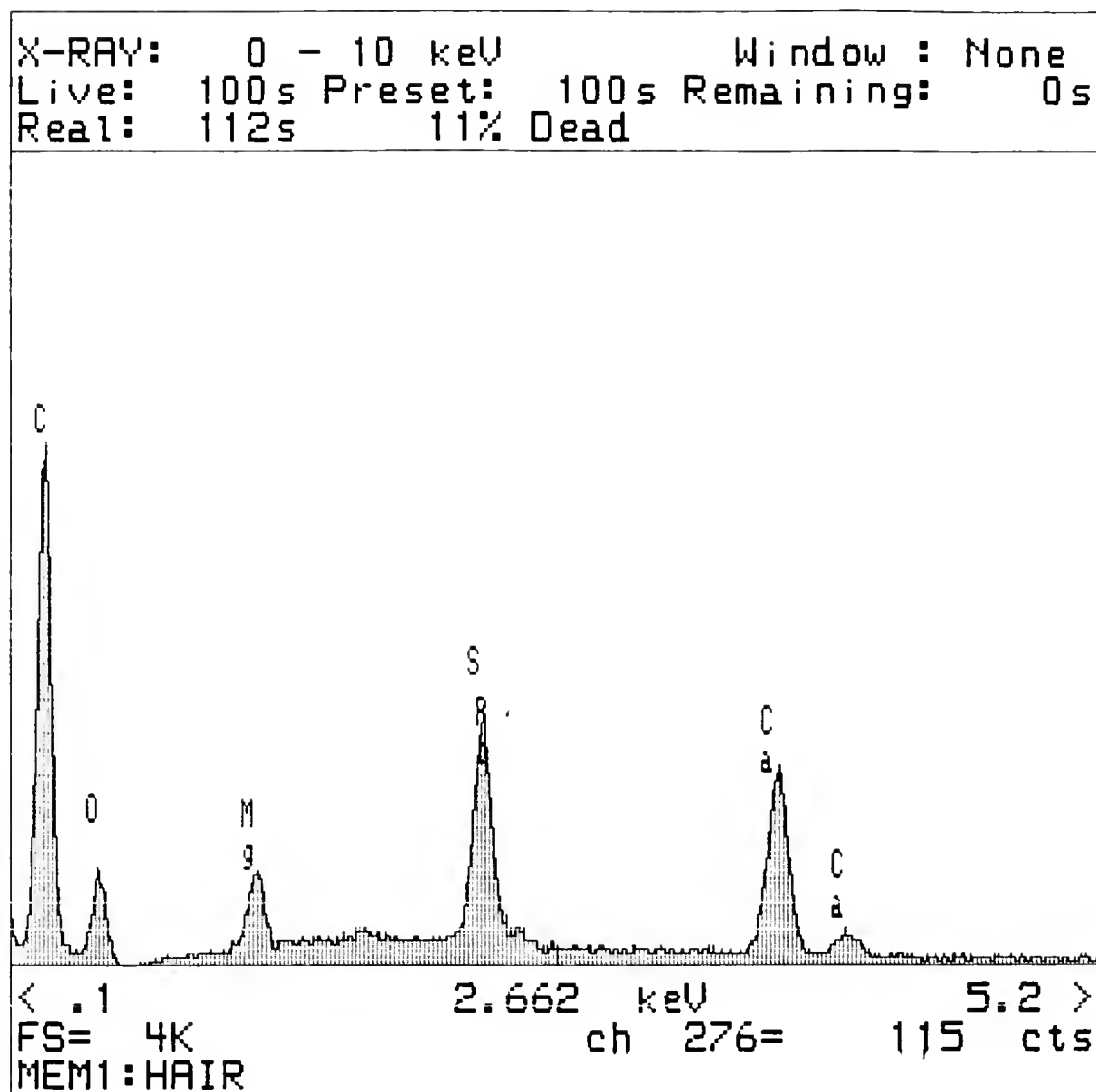




Figure 39: X-Ray Energy Dispersive Analysis of Fiber Found in the Scratch Coat -

Sample 28: Hallway

Presence of sulphur confirms that the fiber is of animal origin. There is no lead in this hair.







#### **4. Paint Failure**

The prevailing condition of paint flaking and powdering can be attributed to moisture as found by Morgan Phillips and Mary Findlay. Contrary to oil based paints, casein paints are porous allowing moisture to evaporate. For good quality casein paint, flaking would not be expected given its porosity. Therefore, the explanation for failure has to be found in the underlying layers. Sizing in particular could act as a moisture barrier especially in the thicker layers. It was found that the sizing was unevenly applied and that flaking coincided with the thicker layers. It is presumed that the gypsum of the finish coat recrystallized below this layer resulting in paint flaking.

Yet another factor may contribute to the phenomena of paint failure. It is the pH difference between the finish coat and the paint layer. Casein becomes soluble at a pH of 4.6. It was found that the plaster finish coat has a pH of 7.97 and the paint layer of 5.67 as measured on a dry sample which showed no signs of paint failure. It is possible, however, that in the presence of moisture, the significant pH difference between the plaster finish coat and the paint layer could cause the casein binder in the paint to dissolve, thus resulting in paint failure. This issue requires further study when treatments for the painted walls are considered.



## 5. Color Alteration

Analysis of pigments used in all the major painted areas in the Lockwood-Mathews Mansion is important to determine the original colors. Pigments are based on chemical compounds and therefore, should obey stoichiometric and kinetic laws of chemical reactions in the course of their deterioration induced by photo-chemical and thermal activity.<sup>102</sup> Fading in sunlight, browning in darkness, and deepening blues over a period of years are all characteristics of some of the pigments used in the nineteenth century.<sup>103</sup> The fading of pigments used in paints is ubiquitous; it is only a matter of time. The more fugitive the pigments used, the shorter the time. As a paint fades, the eye perceives a change in one or more of the dimensions of color such as hue, lightness, and saturation. These subjectively sensed changes are related in a highly complex way to the surroundings of the stimulus area.<sup>104</sup>

Colored pigments for casein should be lime-proof, that is, non-reactive to alkalies. This is the case for the pigments identified in the Lockwood-Mathews Mansion. The principal red, brown and yellow pigments are the iron oxides. Green earth is particularly suitable while zinc green is not as stable; and for a bright green, Malachite can be applied. Ultramarine has been found to be adequate. Also, Indigo and Cobalt blue can be successfully used in alkaline paints. Carbon blacks, black oxide of iron, and manganese dioxide are used for blacks.<sup>105</sup>

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<sup>102</sup>Johnston-Feller, Ruth and Feller, Robert L. *The Kinetics of Fading: Opaque Paint Films Pigmented with Alizarin Crimson and Titanium White*. JAIC, Vol. 23, no. 2, 1984, p. 115.

<sup>103</sup>Findlay, Mary. *Interior Decoration of the Lockwood-Mathews Mansion; Color and Design of the Painted Plaster Walls and Ceilings of the First Floor*. Thesis at Columbia University, 1974, p. 85.

<sup>104</sup>Johnston-Feller, Ruth and Feller, Robert L. *The Kinetics of Fading: Opaque Paint Films Pigmented with Alizarin Crimson and Titanium White*. JAIC, Vol. 23, no. 2, 1984, p. 115.

<sup>105</sup>Sutermeister, Edwin and Browne, Frederick. *Casein and Its Industrial Applications*, New York: Reinhold Pub. Co., 1939, p. 335.



Although color alteration is occurring in the Lockwood-Mathews Mansion, to a great extent, the pigments used are alkali stable. Hence in most cases, it is safe to assume that the paint did not alter dramatically in the last 120 years. It would be of value to monitor further changes in color with the use of a CIE spectrophotometer.

## **Conclusion**

A thorough understanding of the function, techniques, and materials used for the architectural surface is necessary. The analysis of the painted interior finishes of the seventeen rooms in the Lockwood-Mathews Mansion revealed that the paint is a casein based paint. Not only is the paint layer of aesthetical value, but it forms the protective skin of the interior walls. The paint layers are the most vulnerable to entropic effects such as pH, ultra-violet light and moisture. Discoloration is not a major concern in the Lockwood-Mathews Mansion due to the high quality casein paint and correct use of pigments. Paint flaking might be attributed to the varying thickness of the sizing and the pH differential between the finish coat and the paint layer. The research and study of the decorative techniques, materials, and methods of application show an exceptional standard of techniques and quality of materials used in the Lockwood-Mathews Mansion.



## APPENDICES





# APPENDIX A: LIST OF SAMPLES (Fourie)

Rynta Fourie Spring 1997			Box 1
Samples from Lockwood-Mathews Mansion			
Sample #	Location	Specific Location and Details	X-section
1	CII - Washroom of Mrs. Lockwood	ceiling; water damage area	1
2	CII - Washroom of Mrs. Lockwood	ceiling; gold	0
3	CII - Washroom of Mrs. Lockwood	ceiling; overpaint	1
4 A, B	CII - Washroom of Mrs. Lockwood	ceiling; cornice	1
5 A1, A2, A3	CII - Washroom of Mrs. Lockwood	south wall, above lavatories (fragment of design, different colors)	1
6	CII - Washroom of Mrs. Lockwood	ceiling	1
7	CIII - Bathroom of Mrs. Lockwood	ceiling; fragments with red stripes	1
8	CIII - Bathroom of Mrs. Lockwood	ceiling; fragments in grey; some with white stripes	1
9	CI - Mrs. Lockwood's Room	wall (east) exterior band of wall decoration	1
10 A, B	CI - Mrs. Lockwood's Room	wall (east) interior band of wall decoration	1
11	CI - Mrs. Lockwood's Room	wall (east) exterior band of wall decoration	1
12	CI - Mrs. Lockwood's Room	wall (east) from green square of wall decoration; same samples from CII (near lavatories)	1
13	CI - Mrs. Lockwood's Room	wall (east) exterior band of wall decoration	1
14	CI - Mrs. Lockwood's Room	big; wall (north) right side of painting, below electrical outlet	1
15	CII - Washroom of Mrs. Lockwood	window shutter on east wall (some with rot)	1
16	CI - Mrs. Lockwood's Room	east wall; from wood skirting; left side of window light? dark stain barnish on wood??	1
17 A, B, C, D, E, F, G	CII - Washroom of Mrs. Lockwood	south wall; cornice of ceiling; set of seven samples plus extra from cornice above mantel	7
18	CI - Mrs. Lockwood's Room	west wall; left side of door	0
19 A, B	CI - Mrs. Lockwood's Room	ceiling; paint flakes near northwest corner (see sketch)	2
20 A, B, C	CI - Mrs. Lockwood's Room	ceiling; paint flakes near southeast corner (pink stripe from triangle) (see sketch)	3
21	CI - Mrs. Lockwood's Room	ceiling; paint flakes near southeast corner; from retouching (see sketch)	1



22	C1 - Mrs. Lockwood's Room	west wall; set of three samples: #1 (near northwest corner), #2 (center, above door), #3 (near southwest corner) (see sketch)	0
23 A, B	C1 - Mrs. Lockwood's Room	ceiling; paint flakes near southwest corner (see sketch)	2
24	C1 - Mrs. Lockwood's Room	ceiling; paint flakes	1
25	CIII - Bathroom of Mrs. Lockwood	east wall; above window	1
26	CIII - Bathroom of Mrs. Lockwood	south wall; bathroom; different campaigns division?	1
27	CIII - Bathroom of Mrs. Lockwood	bath tub; gold stripes on tub	1
28	Hallway around Rotunda	north wall; decoration near skirting	1
29	R - Rotunda	rotunda, wall around central vestibule (see plan)	1
30 A, B, C	C2 II - Connecting Room to Mrs. Lockwood's Room	various places; set of 3 samples: #1 ceiling, #2 cornice, #3 wall	3
31	C2 II - Connecting Room to Mrs. Lockwood's Room	wall inside closet	0
32	C2 - Mr. Lockwood's Room	wall; near northeast corner	1
33	C2 - Mr. Lockwood's Room	wall; east wall near window	1
34	C2 - Mr. Lockwood's Room	east wall; touch up ?? (sample composed of flakes)	1
35 A	C2 IV - Bathroom of Mr. Lockwood's	wall; near radiator	1
36	C2 IV - Bathroom of Mr. Lockwood's	wall; two green campaigns; inside closet	1
37	C3 - Moorish Room	wall; above door (see map)	1
38 A, B	C3 - Moorish Room	wall; above door (see map) (flakes)	2
39	C2 I - Storage Room of Mr. Lockwood's	wall	1
40	Italian Suite	painted wood; green birch eye	1
41	Secondary Staircase, Third Floor	blue stripe staircase; third floor	1
42	M - Music Room	wall near northeast corner, brown flower	1
43	M - Music Room	field; problem area	1
44 A, 44 A2	M - Music Room	green	1
45 A	M - Music Room	blue thin line	1
46	M - Music Room	gold	1
47	M - Music Room	gold and leaves	1
48	M - Music Room	wood door	1
49	M - Music Room	beige	1
50	M - Music Room	brown flower	1
51 A, B	R - Rotunda	gold and field	2
52	R - Rotunda	vermillion	1



53	R - Rotunda	weird with gold	1
54	R - Rotunda	unknown impasto	1
55	L - Library	graining	1
56	L - Library	gold	1
57	Unknown	green	0
58	M - Music Room	orange/red-brown	1
59	M - Music Room	field; sound area	1
60	M - Music Room	field; problem area	1
61	C3 - Moorish Room	field	1
62	Mirror Room	field	1
63	M - Music Room	field	1
64	Wash Room	first campaign	1



## APPENDIX B: MUSIC ROOM

**Figure 40: Photograph of Design in Music Room**

**Type of Film:** 200 ASA Kodak Gold

**Camera:** Minolta

**Comments:** Numbers indicate where some samples were taken on the wall. The orange, blue line, green leaves, brown, gold, and beige inside the border and the field were tested.







**Figure 41: Photo Micrograph of Sample 42: Music Room**

**Sample Location:** Music Room: Brown Flower

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 12A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



Light brown layer  
part of design.

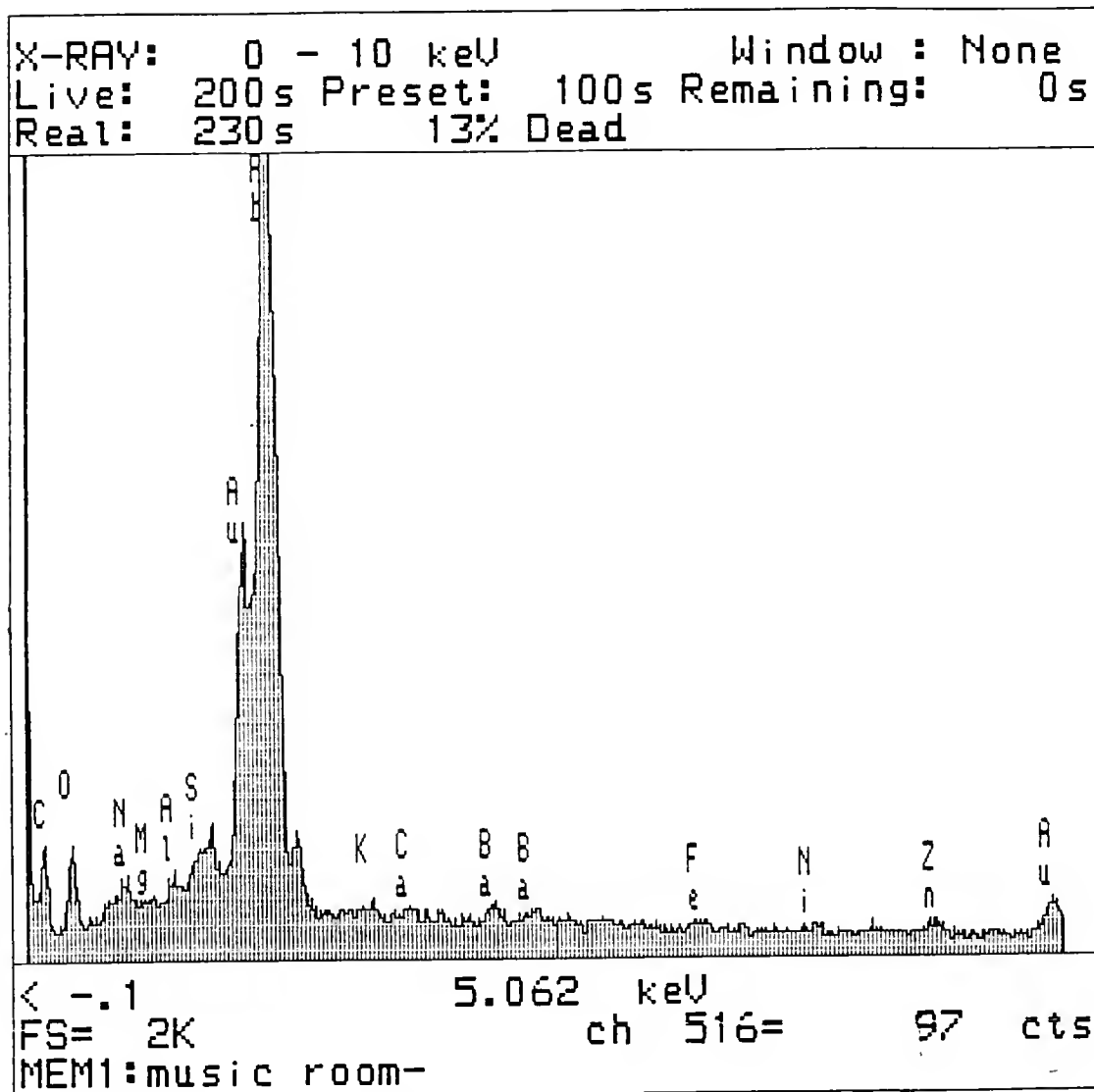
Base coat

— Gypsum substrate



**Figure 42: X-Ray Energy Dispersive Analysis of Sample 42: Music Room**

This is a gold coated sample. The brown color does not reveal mercury: an indication of the presence of vermillion. It is possible that there is green earth, iron oxides and even some blue pigments such as ultramarine. Lithopone is present.





**Figure 43: Photo Micrograph Sample 43: Music Room**

**Sample** 43

**Sample Location:** Music Room Field

**Type of Film:** 200ASA Kodak Royal Gold, Film 3 Negative 12A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

**Comments:** Sample from the field of the Music Room. The major color in the room in this room is referred to as lavender (Fig 9). There is no calcite or quartz particles in the finish coat.



Paint Layer with  
blue pigment  
particles.

Yellowish Primer.

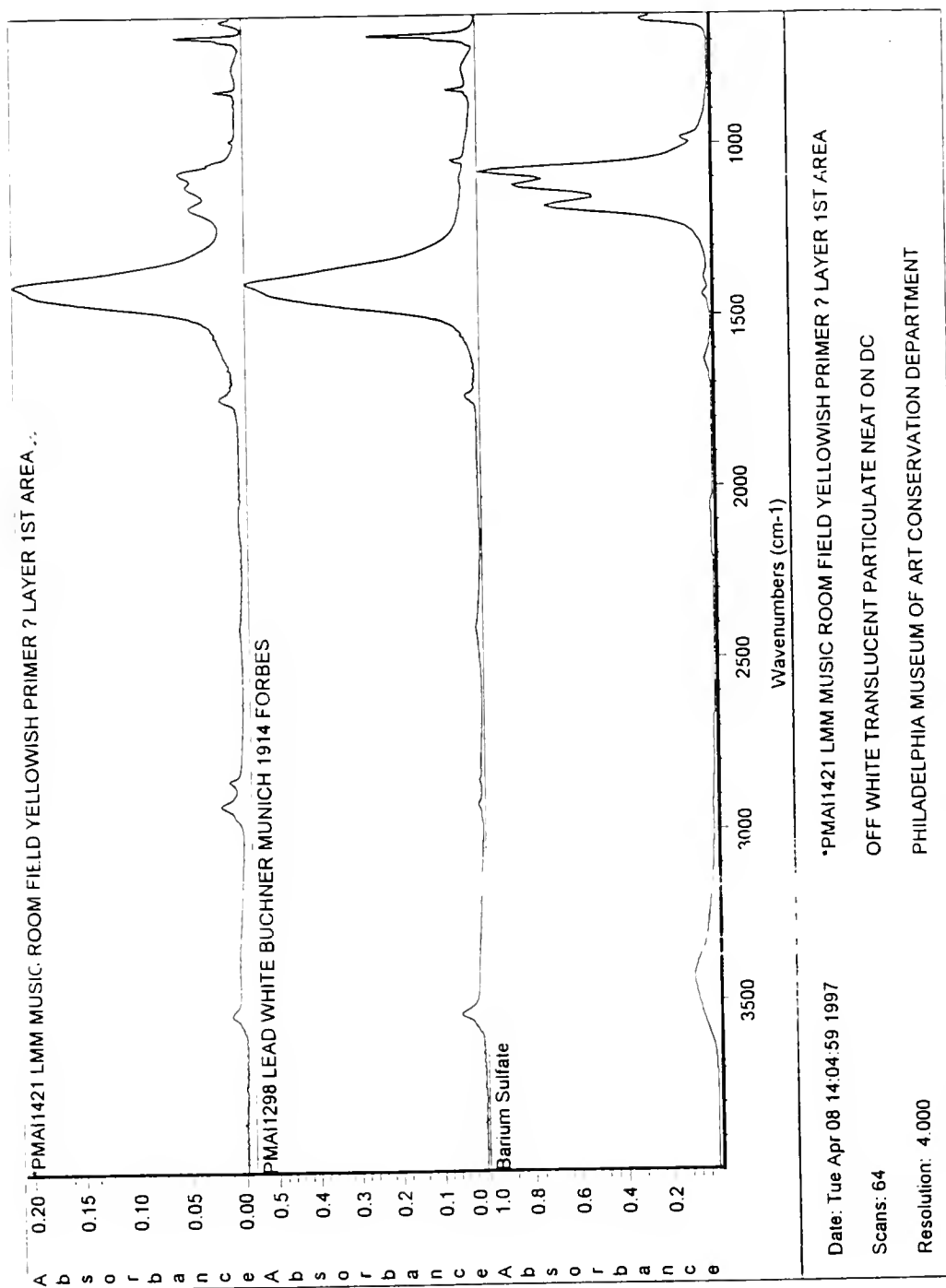
Sizing is not clearly  
visible. (Fig. 9)

**Tests:** FT-IR shows binder positive for casein and/or animal hide glue. Nepheline Chloride spot test was done. It indicated no presence of oil or wax. FT-IR identified lead white and baryite in the paint. No red lead was identified. This pigment might have been used to make the lavender-like color. It seems that Alizarin Crimson was used.



**Figure 44: FT-IR of Sample 43: Music Room**

Analysis of the paint layer. Lead White and Barium Sulfate are identified. The bottom graph is the sample and the two top graphs are the standards.







**Figure 45: FT-IR of Sample 43: Music Room**

Top two graphs compare the yellowish primer and the top paint layer. The two paint layers are very similar in composition; however, there is a small difference. (Fig. 44)

The bottom graph is the finish coat. It is gypsum.

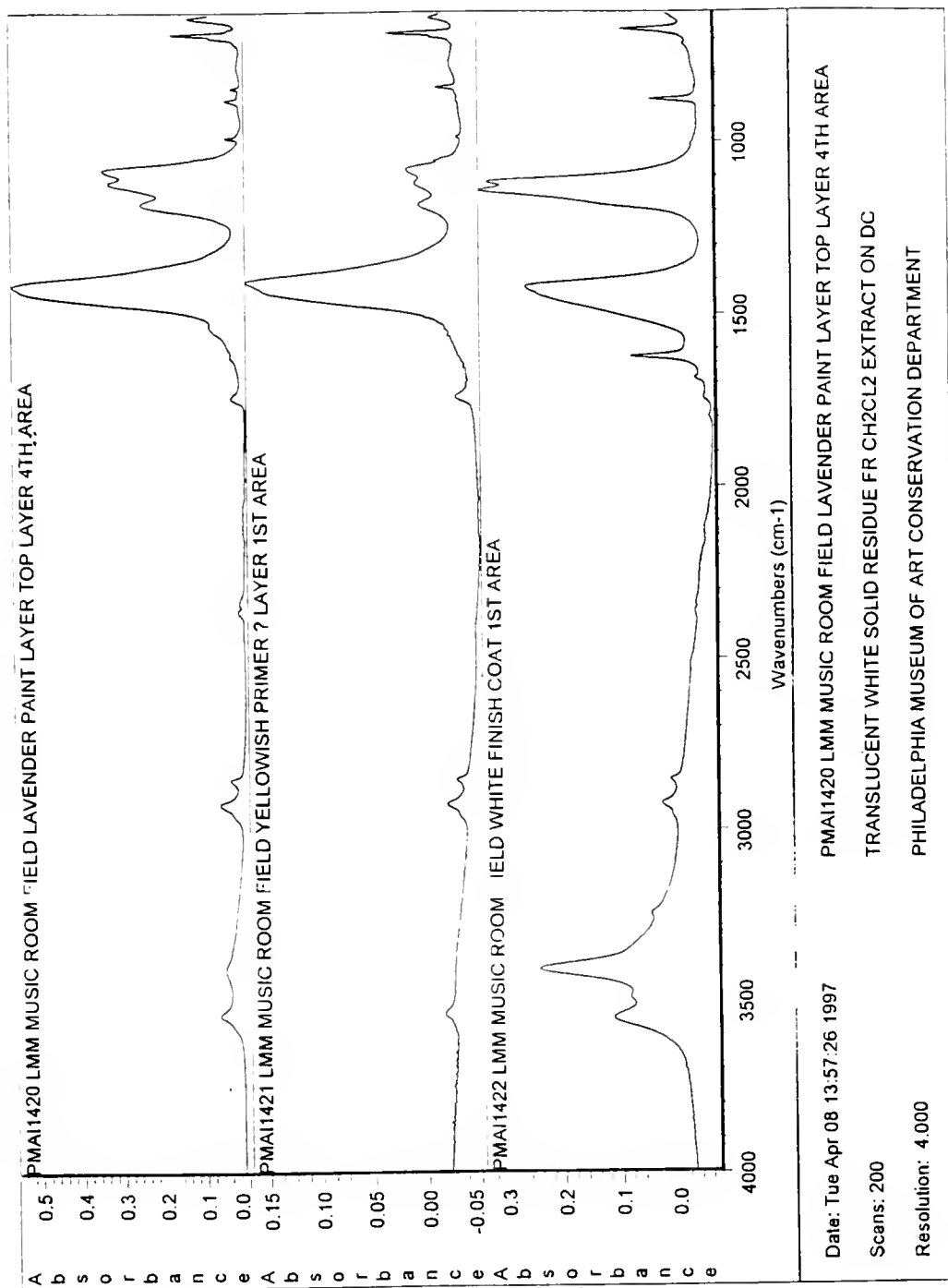
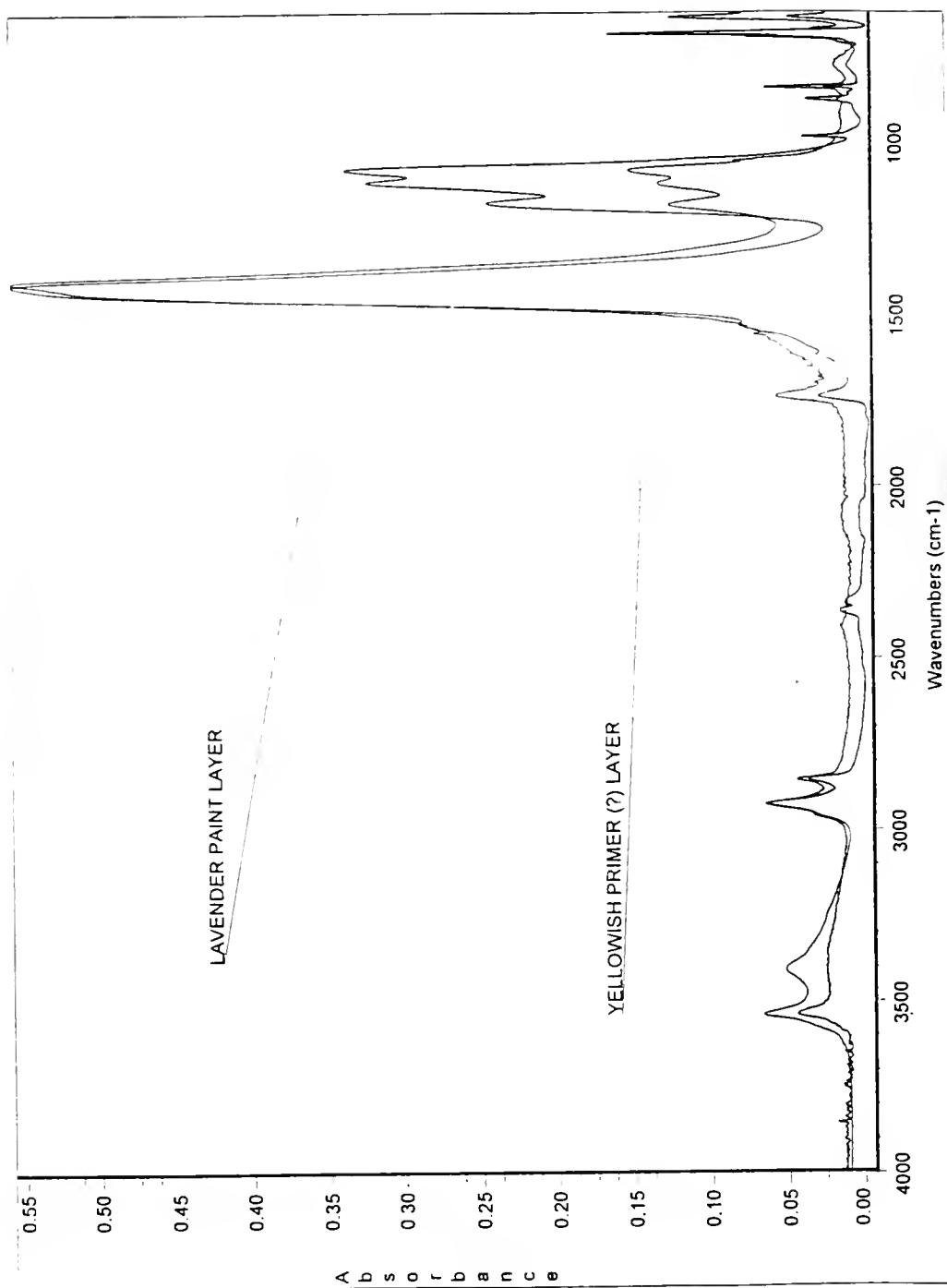




Figure 46: FT-IR of Sample 43: Music Room

Comparison between the primer and the top paint layer.





**Figure 47: Photo Micrograph of Sample 44: Music Room**

**Sample Location:** Music Room - Green leaves

**Type of Film:** 200 ASA Kodak Royal Gold, Film 5 Negative 12

**Camera:** Nikon

**Magnification:** 225X

**Reflected light**



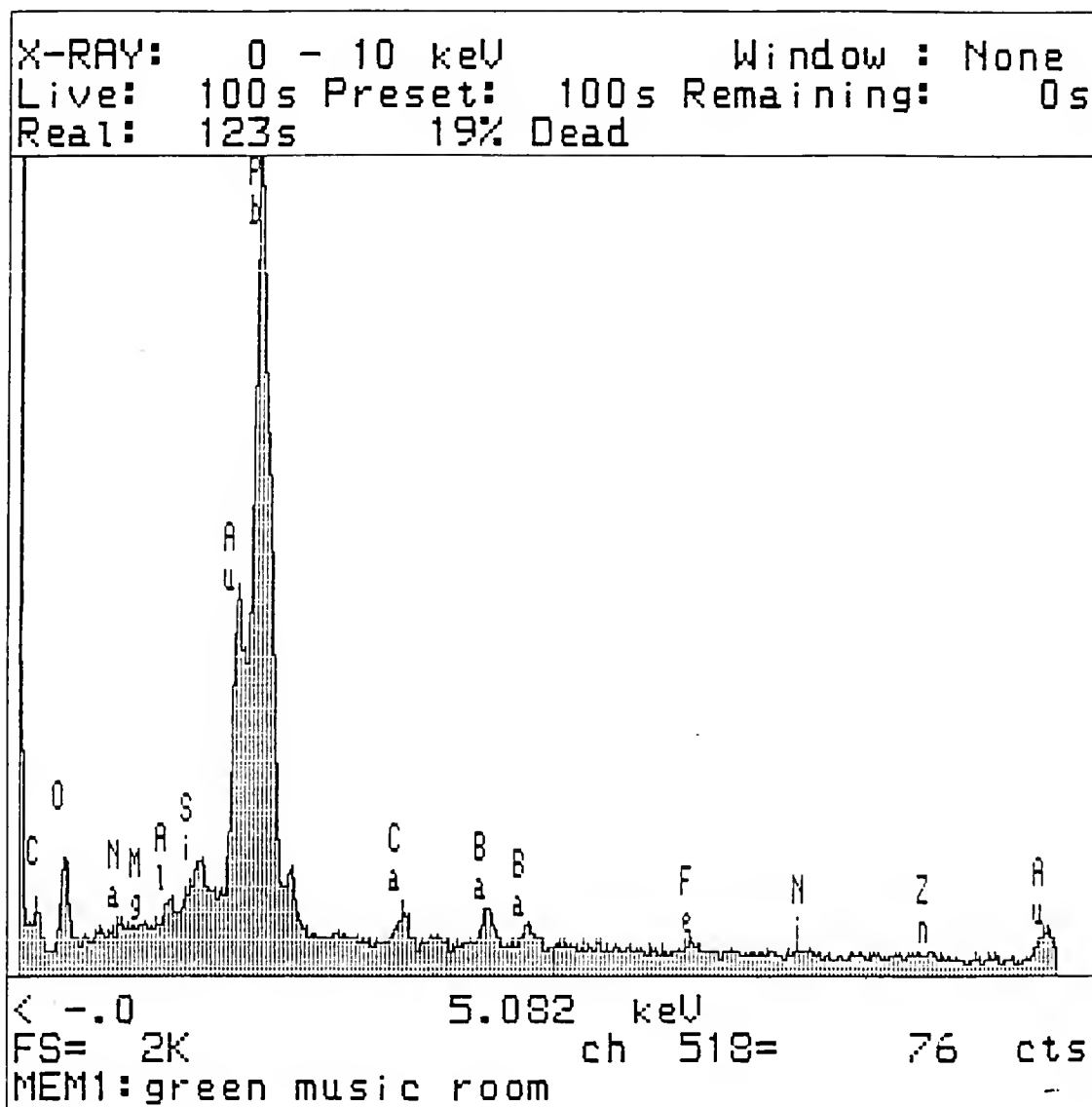
— Green paint layer,  
green pigments are  
more concentrated  
near the surface.

— Substrate



**Figure 48: X-Ray Energy Dispersive Analysis of Sample 42: Music Room**

This gold coated sample revealed that the green was made with green earth and ultramarine. The rest of the chemical compounds are lithopone.







**Figure 49: Photo Micrograph of Sample 45: Music Room**

**Sample Location:** Music Room - Blue Line

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 15A

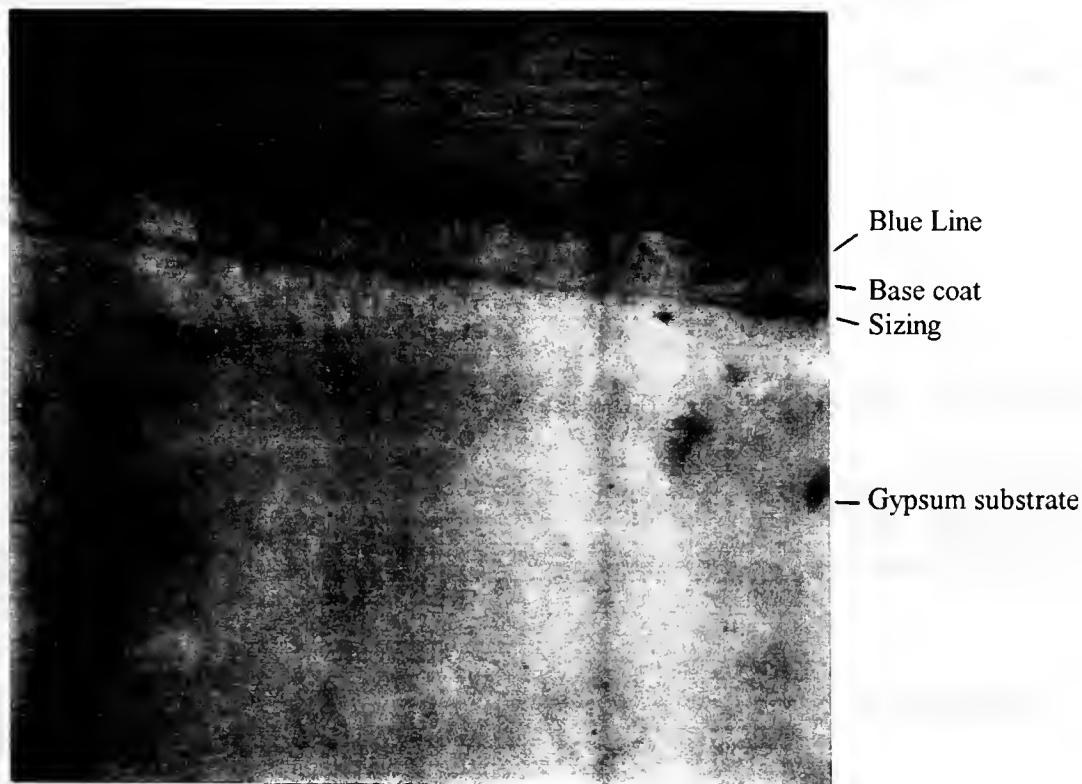
**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

**Comments:** It looks like the same blue pigments are in the blue line as in the field - just more blue. These pigments were identified as ultramarine. The blue line is to the left of the micrograph. It looks like the line was painted shortly after the field color was applied.

**Tests:** Micro chemical identification of pigments spot tests revealed that no Prussian blue was present. The ultramarine test gave a reaction, but it was influenced by the presence of lithopone in the paint. FT-IR indicated a suggestive presence of ultramarine.





## Figure 50: Photo Micrograph of Sample 46: Music Room

**Sample Location:** Music Room - Gold

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 16A, Film 5 Negative 18

**Camera:** Nikon

**Magnification:** 125X and 225X

### Reflected light

**Comments:** No gold is identified but copper and silver are. It seems to be a glazing technique. The results of the X-Ray energy dispersive analysis of this sample with gold and carbon coat contradict each other.



- Paint layer with gold  
Base coat, lavender
- Sizing
- Gypsum substrate

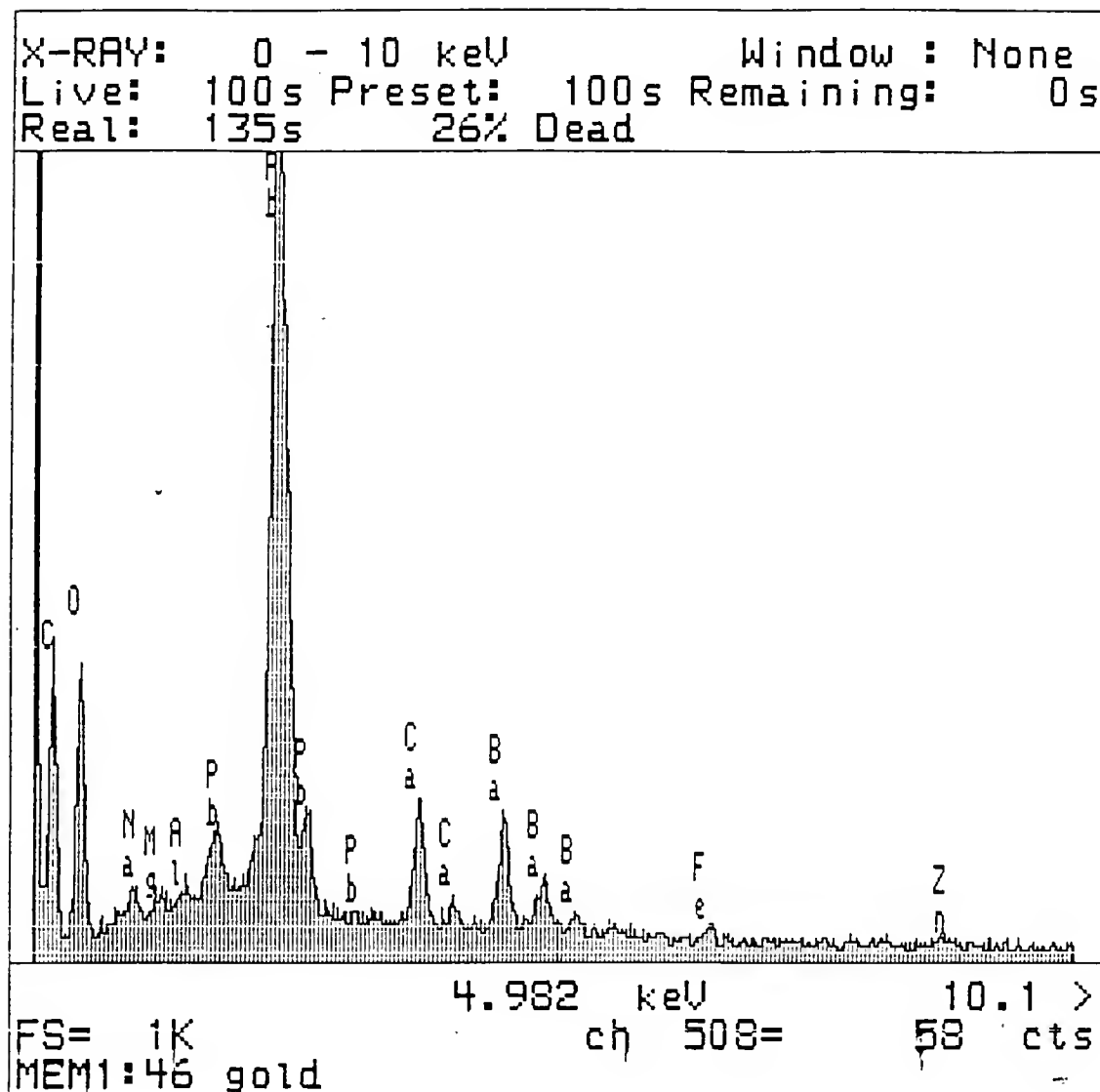


- Paint layer with gold
- Base coat, lavender  
with blue and red  
pigment particles
- Gypsum substrate



**Figure 51: X-Ray Energy Dispersive Analysis of Sample 46: Music Room**

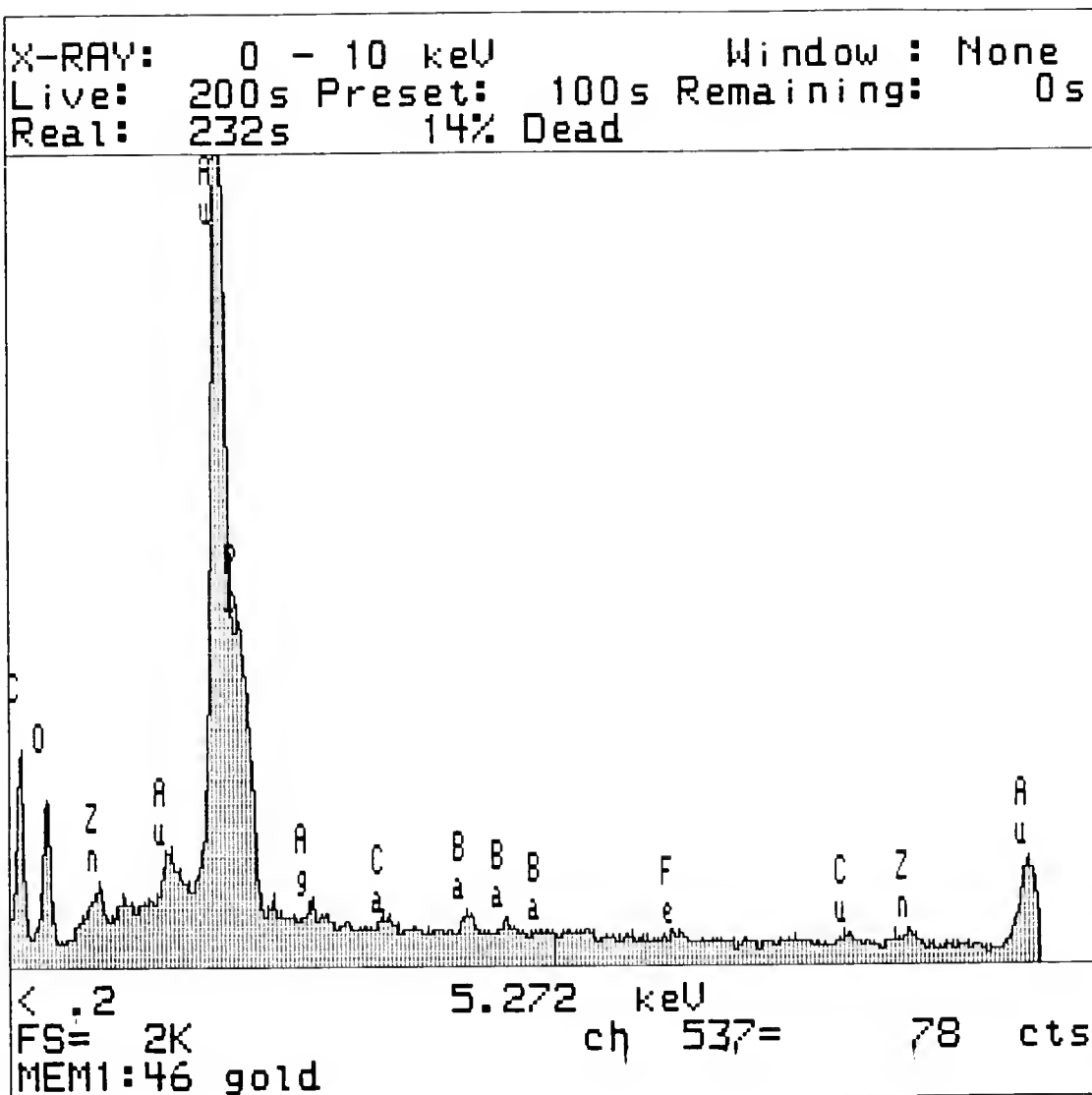
This sample was carbon coated. It revealed no gold, silver or copper. But the paint layer has calcium sulfate, lead oxide, and iron oxides such as yellow ochre. The high quantity of lead and the presence of zinc and barium indicates that lithopone is present. However, the analyses of the same sample but coated with gold revealed silver and copper.





**Figure 52: X-Ray Energy Dispersive Analysis of Sample 46: Music Room**

This sample was coated with gold. Contrary to the results in figure 51, silver and copper was found. It is not possible to say if this is gilding in comparison to the gold leaf of the Moorish Room and the Bathroom of Mrs. Lockwood's Room. However, it does not seem to be metal leaf, but rather powder. Tests also confirm the presence of lithopone and iron oxides which suggests that the layer is paint.







**Figure 53: Photo Micrograph of Sample 47: Music Room**

**Sample Location:** Music Room - gold and leaves

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 17A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

**Comments:** The gold layer is very thin.





**Figure 54: Photo Micrograph of Sample 47: Music Room**

**Sample Location:** Music Room - gold and leaves

**Type of Film:** 200 ASA Kodak Royal Gold, Film 4 Negative 7

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

**Comments:** Positive for proteins tested with FITC





**Figure 55: Photo Micrograph of Sample 49: Music Room**

**Sample Location:** Music Room - beige inside border

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 18A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

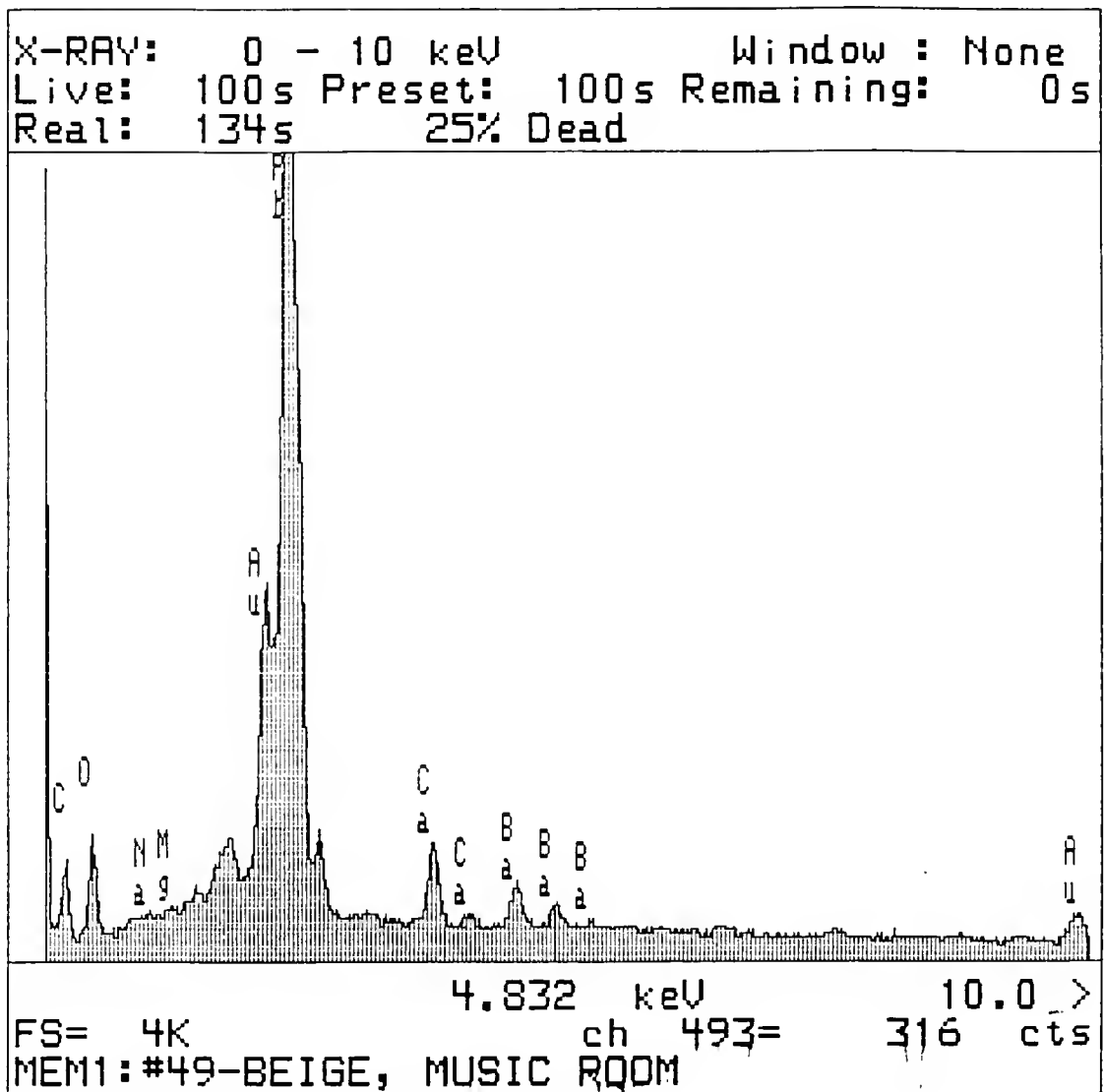
**Comments:** Seems to be similar as the field, just less pigment.





**Figure 56: X-Ray Energy Dispersive Analysis of Sample 49: Music Room**

This sample was gold coated. Lead white, baryite, and carbon blacks were found, but no lithopone was found in this sample.

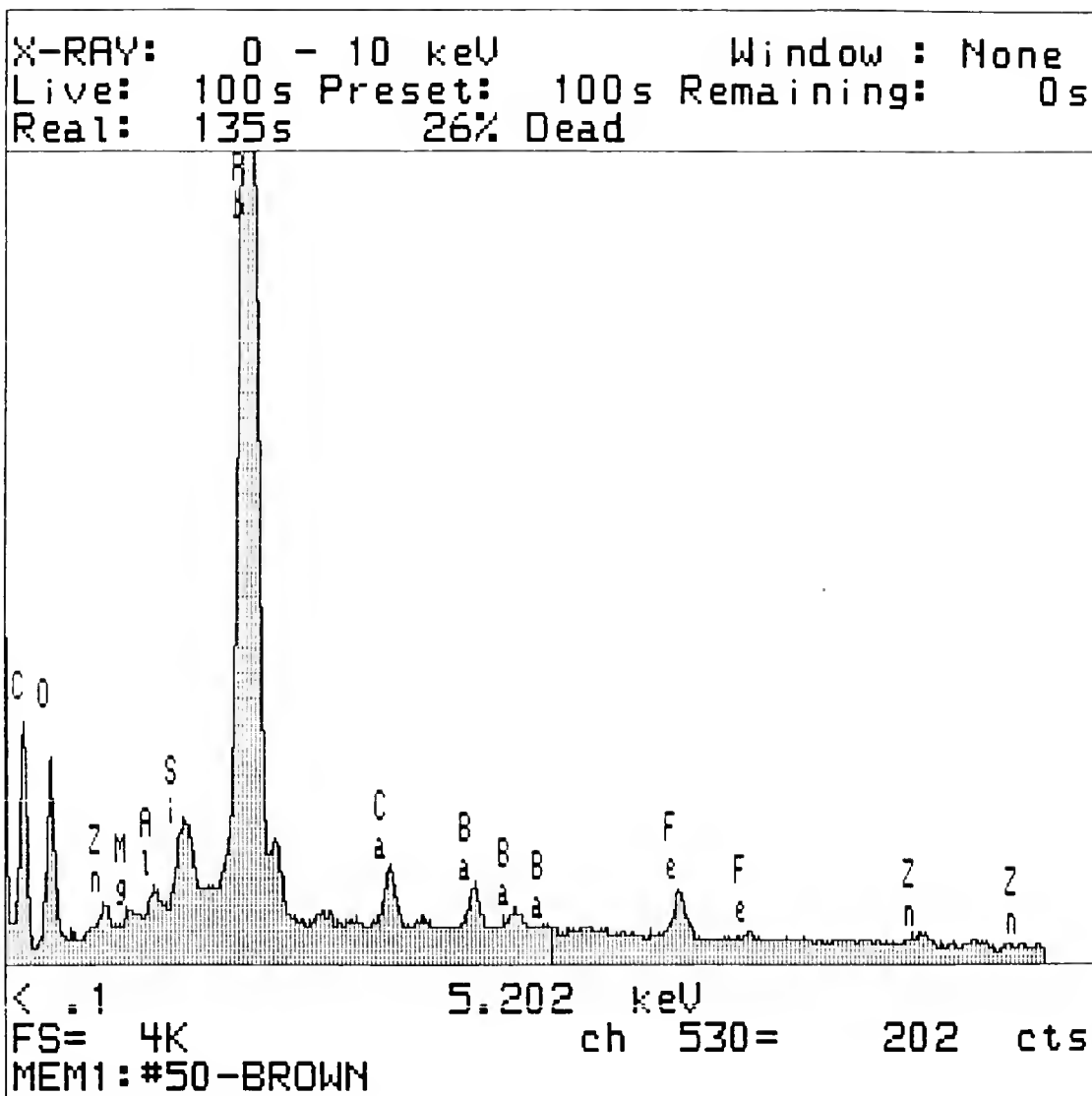






**Figure 57: X-Ray Energy Dispersive Analysis of Sample 50: Music Room**

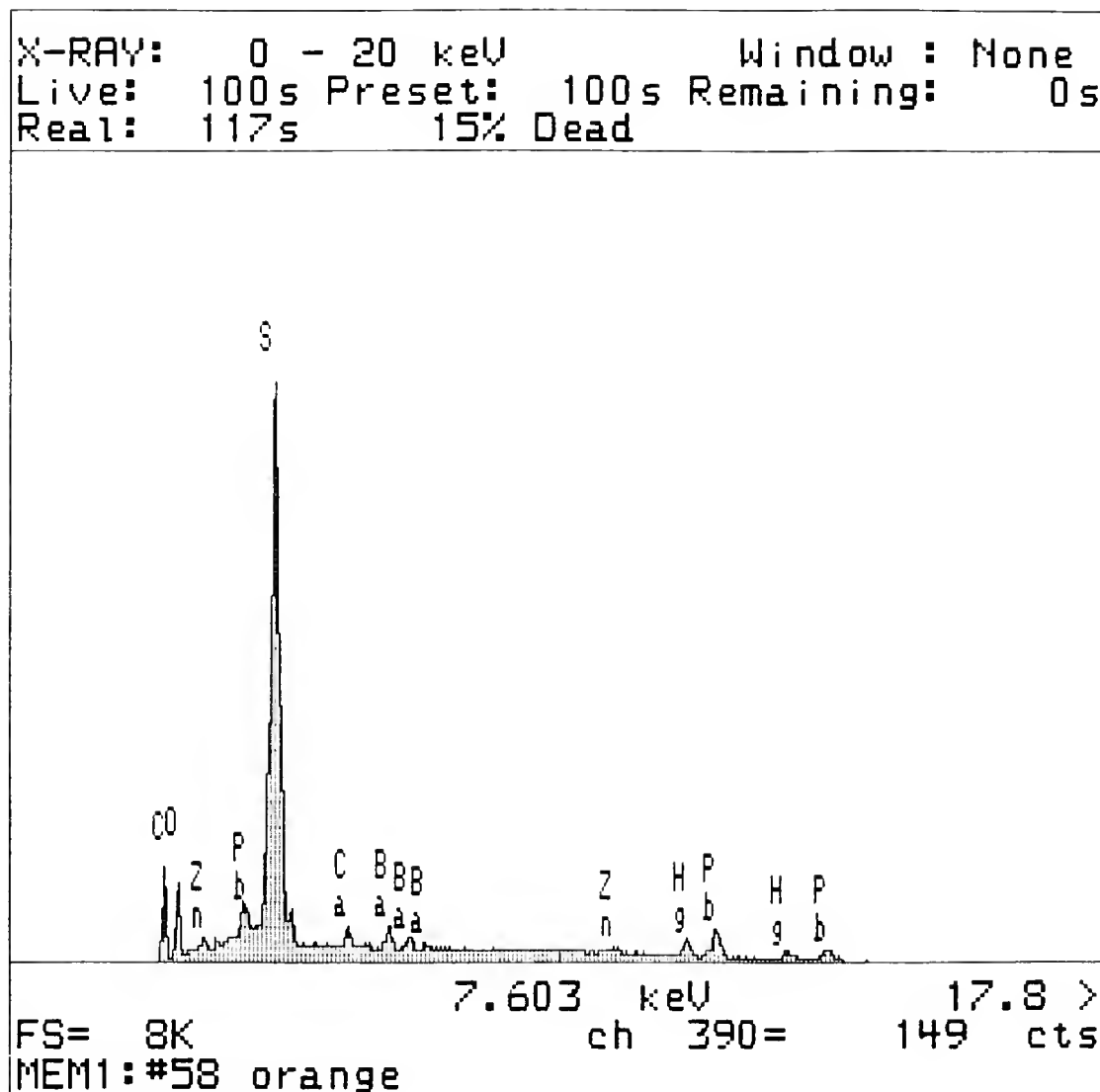
This sample was carbon coated. The paint color is brown. It contain carbon black, iron oxide and lithopone. The magnesium, aluminum, and silica might be part of the part of burnt sienna or umber. (Fig. 11)





**Figure 58: X-Ray Energy Dispersive Analysis of Sample 58: Music Room**

This carbon coated sample indicated that the orange paint is vermillion. Lithopone is present, but lead does not show because the sulphur peak overlaps. It seems that calcium carbonate is also present.





**Figure 59: Secondary Electron Image of Sample 60: Music Room**

This sample is carbon coated. The sample is from the field where the paint failure is present. A study was made of the moisture damaged and undamaged areas. X-Ray energy dispersive analyses were done on the dark and light areas of the secondary electron image. It indicates that gypsum recrystallises below the paint layer.

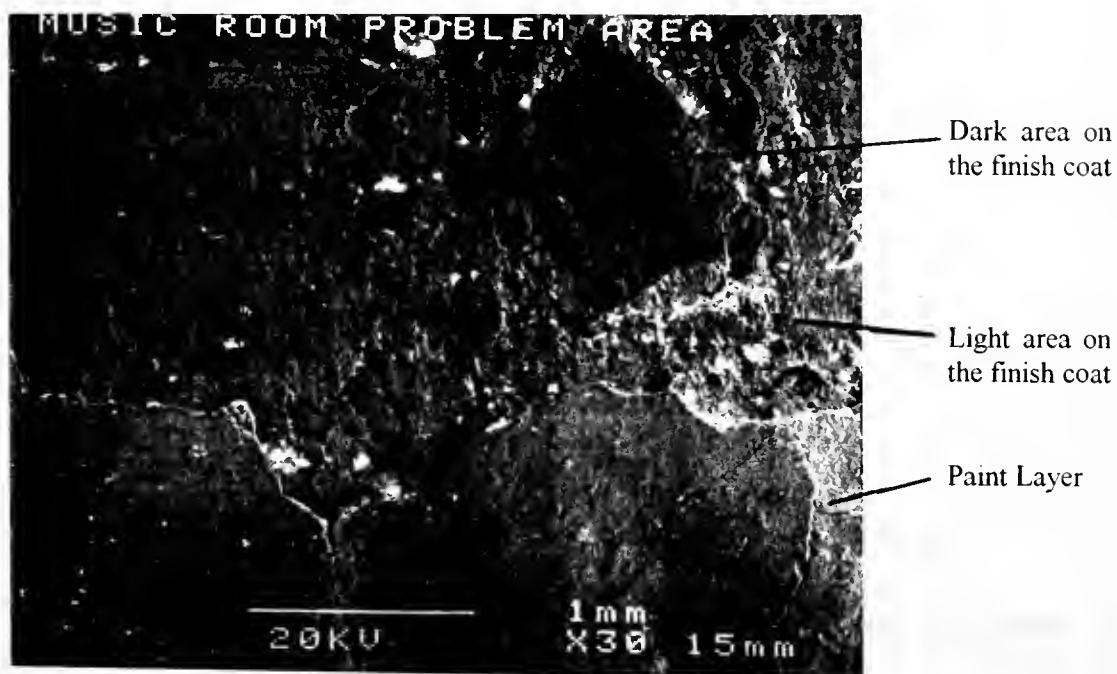




Figure 60: X-Ray Energy Dispersive Analysis of Sample 59: Music Room

The dark area as indicated in figure 59 shows gypsum crystals are forming. Mostly sulphur is present - no lead.

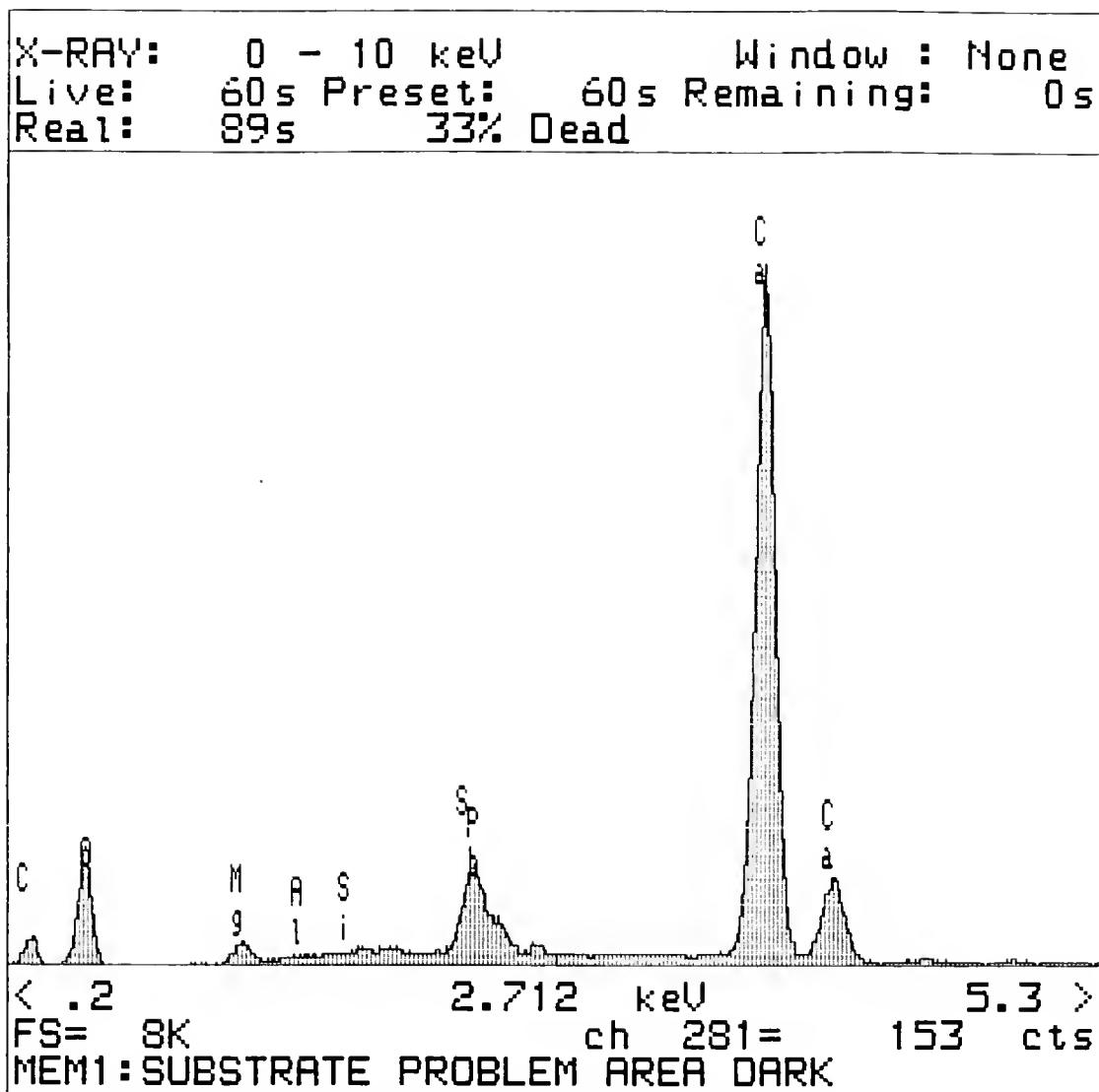
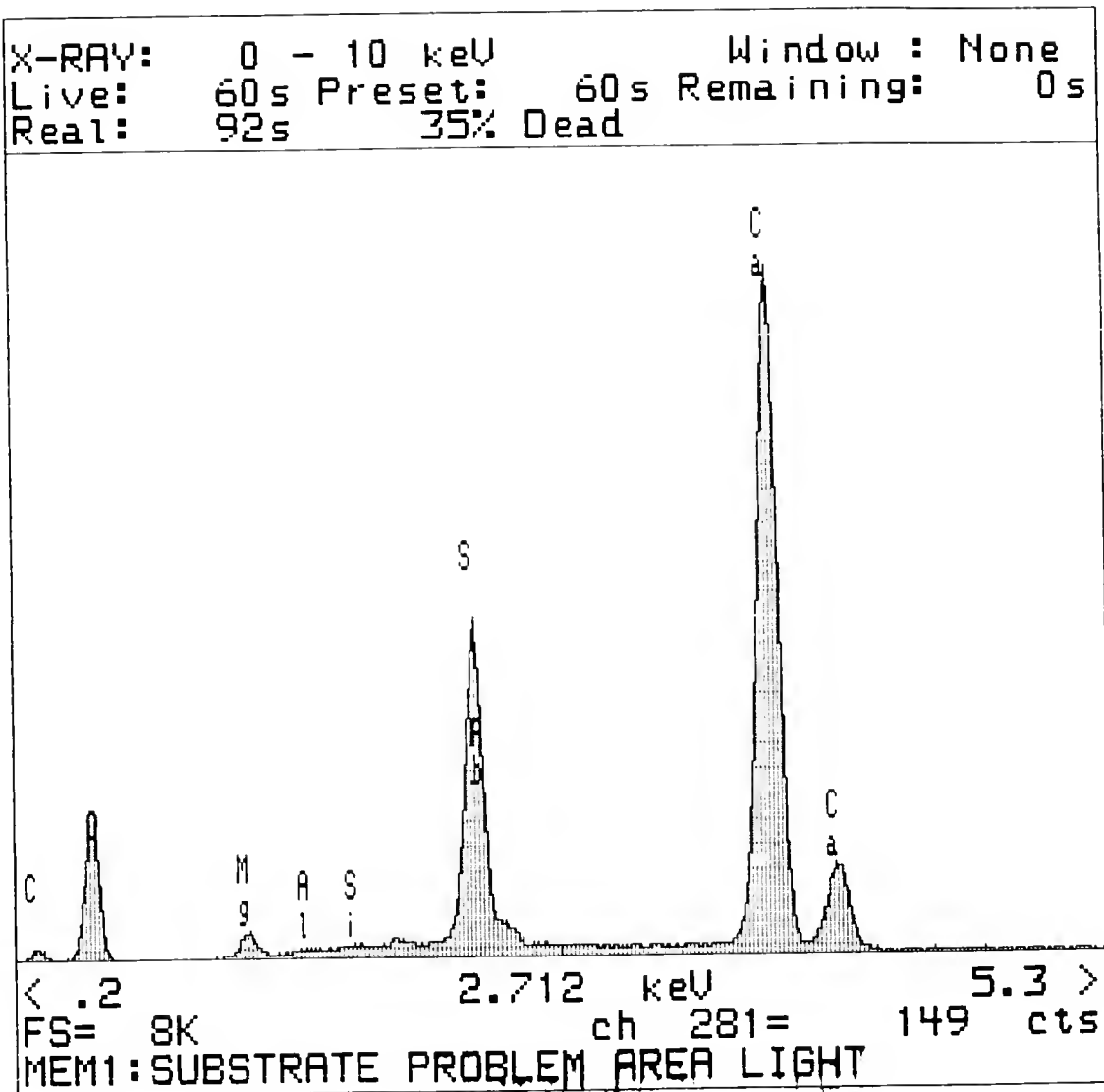






Figure 61: X-Ray Energy Dispersive Analysis of Sample 59: Music Room

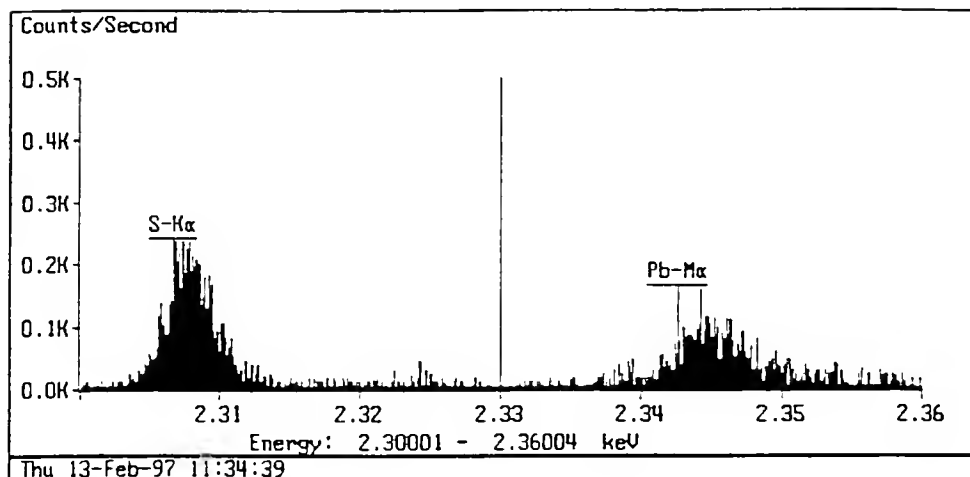
The light area has lead and sulphur. The higher presence of sulphur indicates that the gypsum recrystallizes.





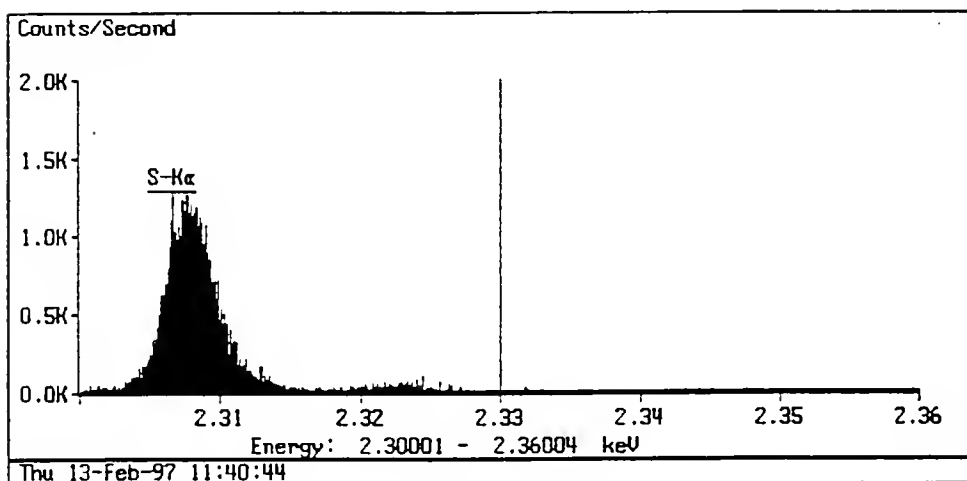
**Figure 62: Wavelength Dispersure Spectrometer of Sample 59: Music Room**

Dark area indicates that lead and sulphur is present.



**Figure 63: Wavelength Dispersure Spectrometer of Sample 59: Music Room**

Light area indicates that sulphur is present but no lead.





## APPENDIX C: MRS. LOCKWOOD'S ROOMS

**Figure 64: Photographs of Mrs. Lockwood's Room**

This room has a washroom and bathroom and a connecting room to Mr. Lockwood's Room. The washroom and Mrs. Lockwood's Room are the same design and technique. The connecting room is similar in paint and materials but has no designs.

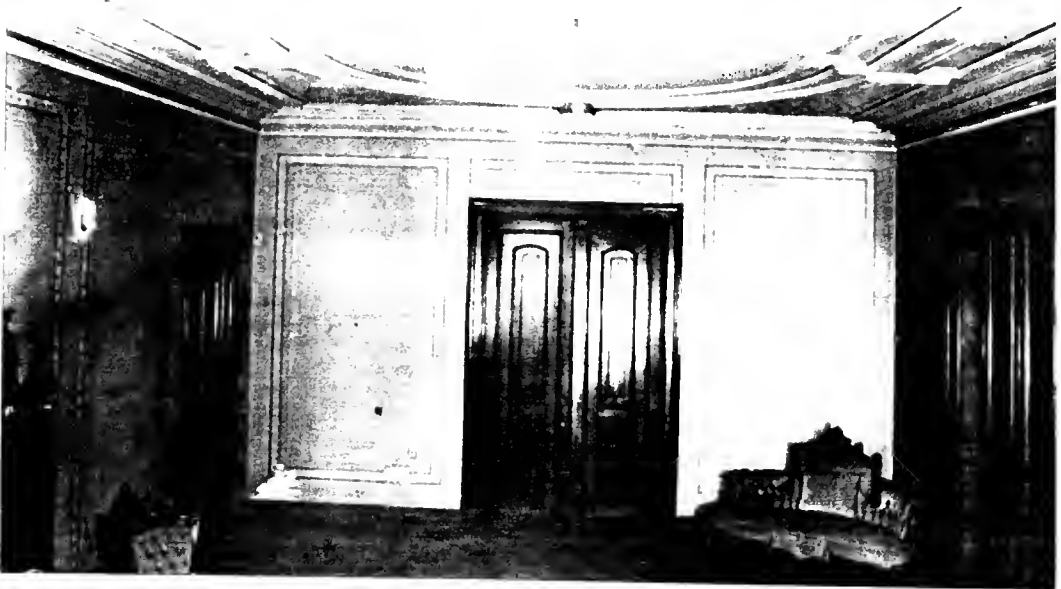
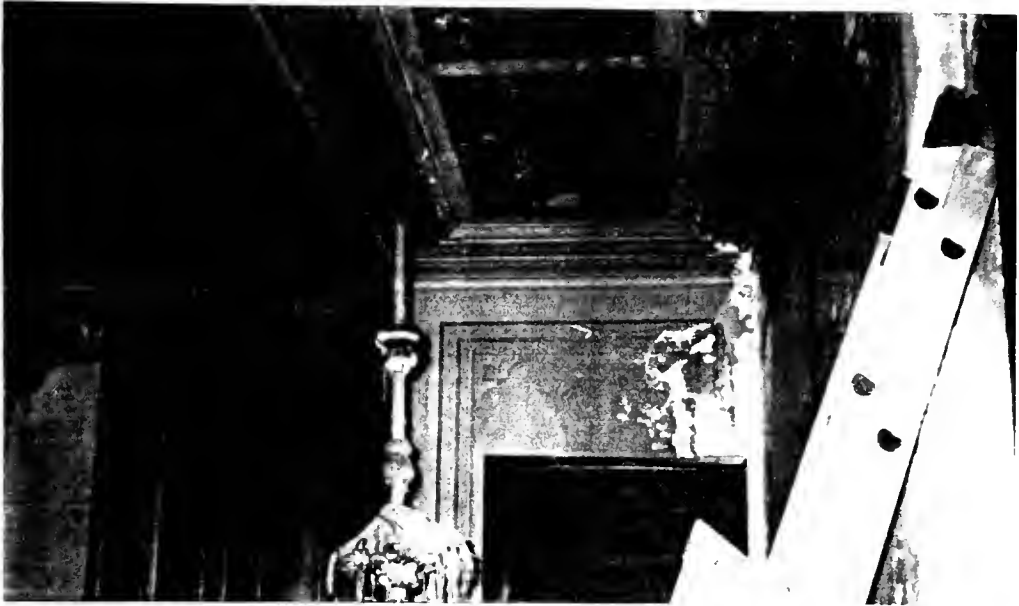




Figure 65: Photographs of Mrs. Lockwood's Washroom







**Figure 66: Photograph of Bathroom of Mrs. Lockwood's Room**





**Figure 67: Photograph of Sample 17 area: Washroom of Mrs. Lockwood's Room**

Seven samples were taken on the moulding near the ceiling of the south wall. They are 17A-G. Just below the cornice is a blue line, this is Sample 1.





**Figure 68: Photo Micrograph of Sample 17A: Washroom of Mrs. Lockwood's Rm**

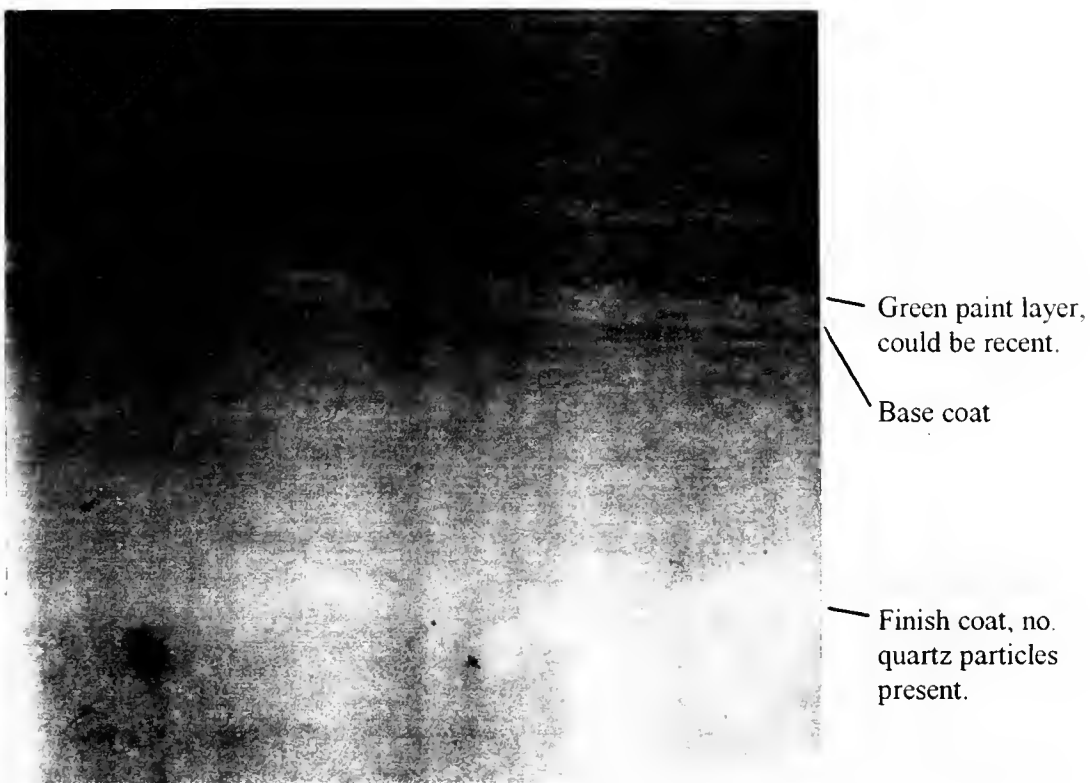
**Sample Location:** Cornice at ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 11

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 69: Photo Micrograph of Sample 17B: Washroom of Mrs. Lockwood's Rm**

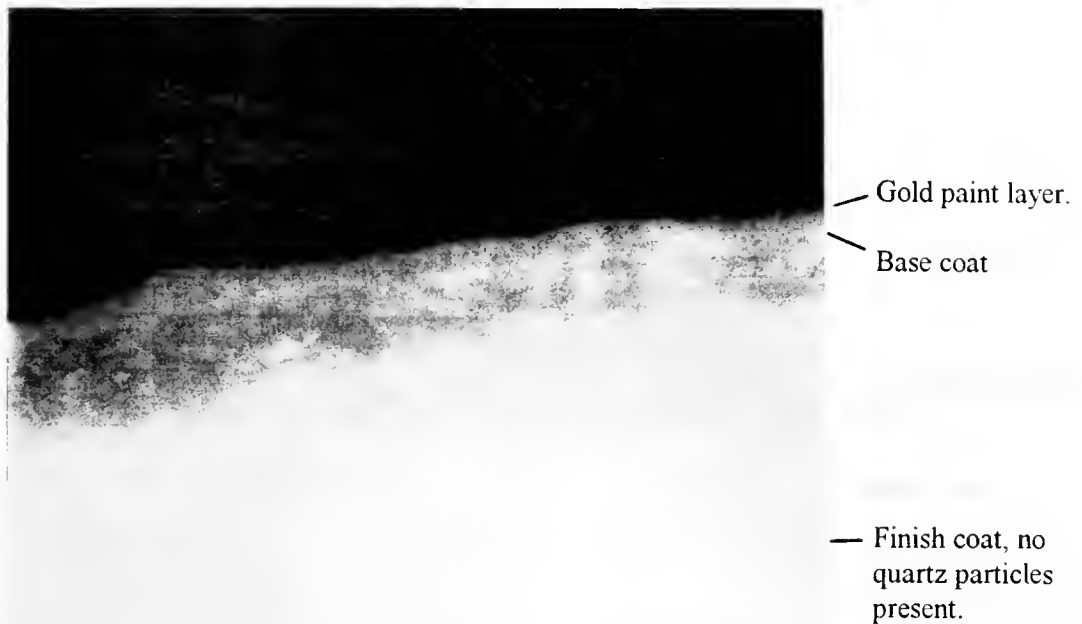
**Sample Location:** Cornice at ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 12

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**







**Figure 70: Photo Micrograph of Sample 17C: Washroom of Mrs. Lockwood's Rm**

**Sample Location:** Cornice at ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 13

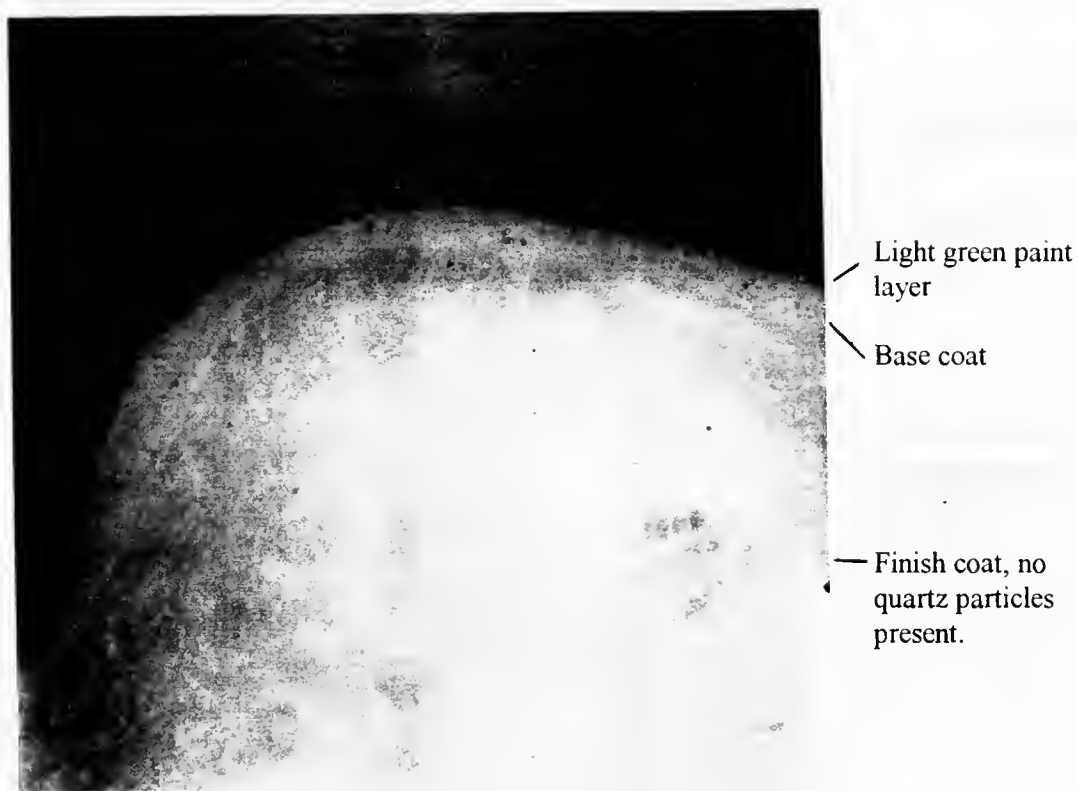
**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

**Comments:** Cornice at ceiling of Washroom

Sample 17C and D are the same.





**Figure 71: Photo micrograph of Sample 17D: Washroom of Mrs. Lockwood's Rm**

**Sample Location:** Cornice at ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 14

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



Light green paint  
layer.

Base coat

Sizing

Finish coat, no  
quartz particles  
present.



**Figure 72: Photo Micrograph of Sample 17E: Washroom of Mrs. Lockwood's Rm**

**Sample Location:** Cornice at ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 15

**Camera:** Nikon

**Magnification:** 225X

**Reflected light**



Green paint layer,  
could be recent

Base coat with a  
white primer?

Sizing

Finish coat, no  
quartz particles  
present.



**Figure 73: Photo Micrograph of Sample 17F: Washroom of Mrs. Lockwood's Rm**

**Sample Location:** Cornice at ceiling of Washroom

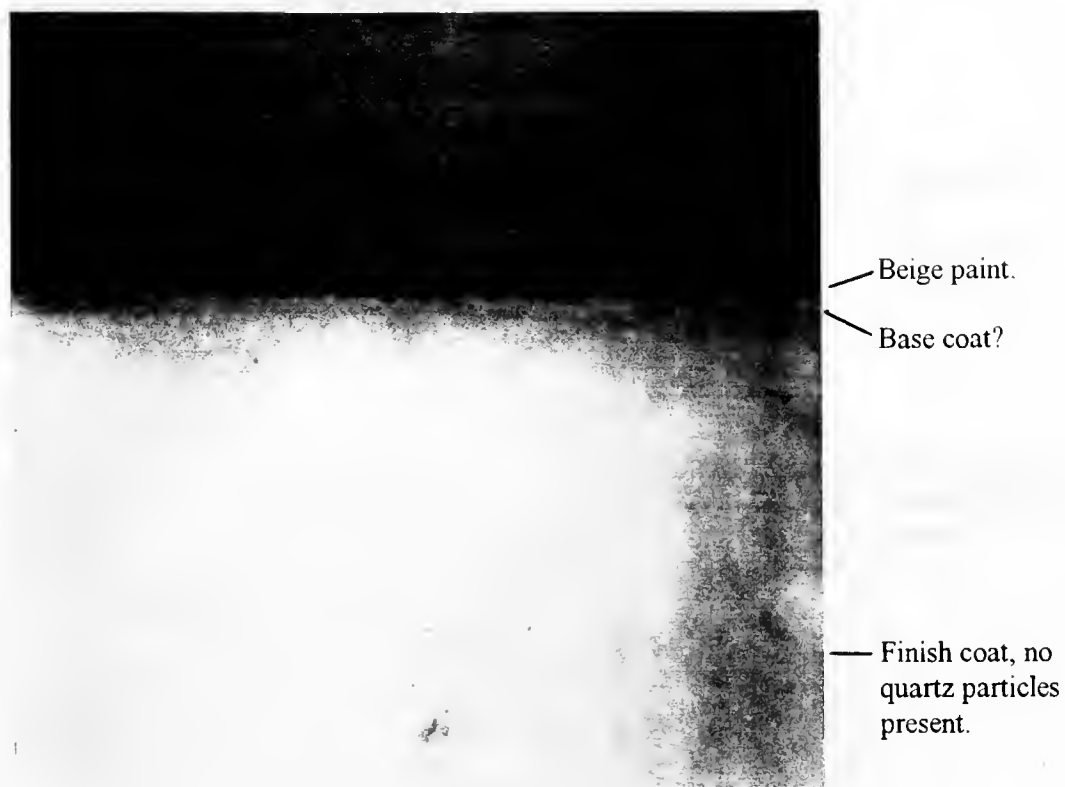
**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 16

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

**Comments:** It seems that Sample 17F and G are similar.







**Figure 74: Photo micrograph of Sample 17G: Washroom of Mrs. Lockwood's Rm**

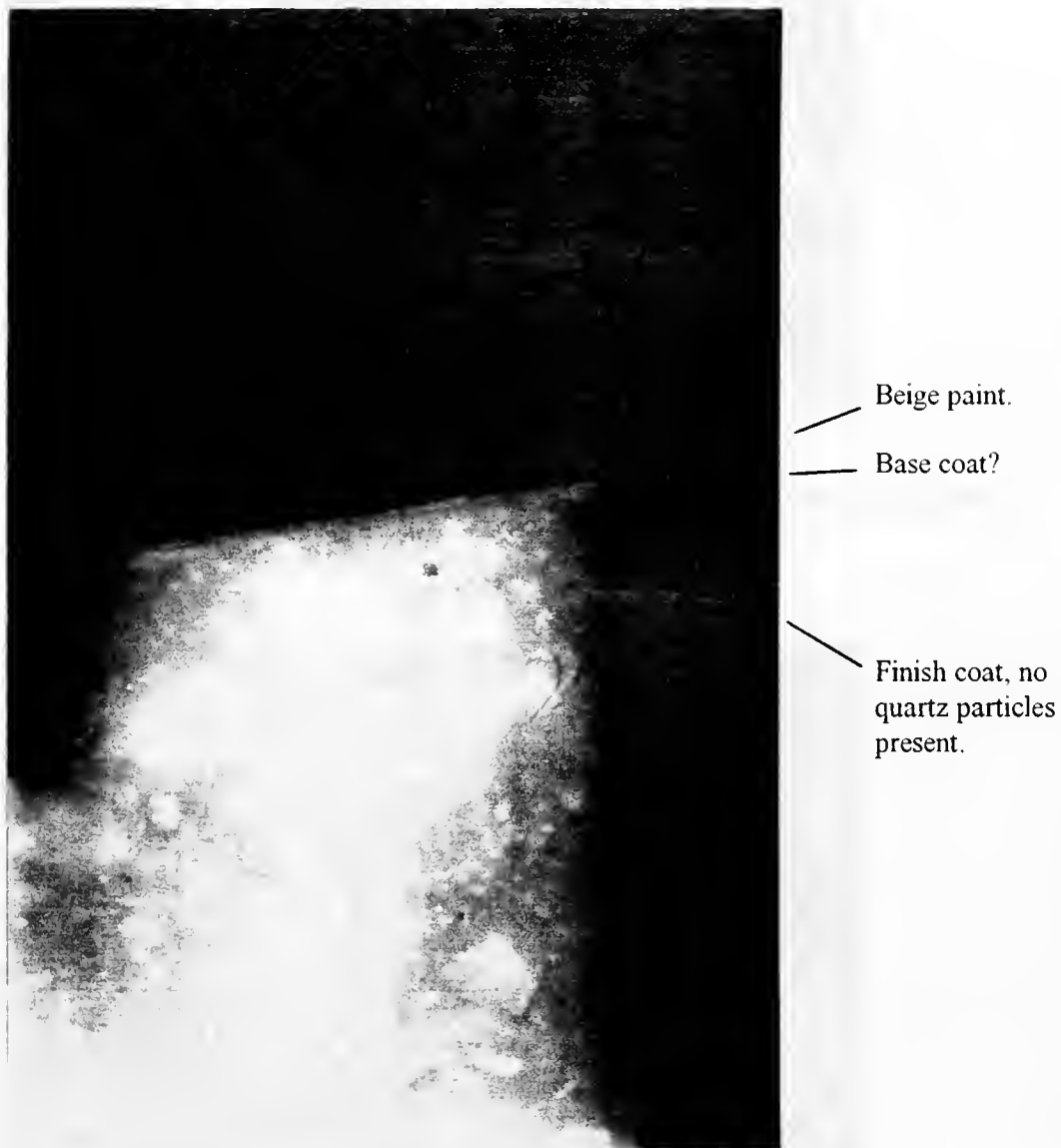
**Sample Location:** Cornice at ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 17

**Camera:** Nikon

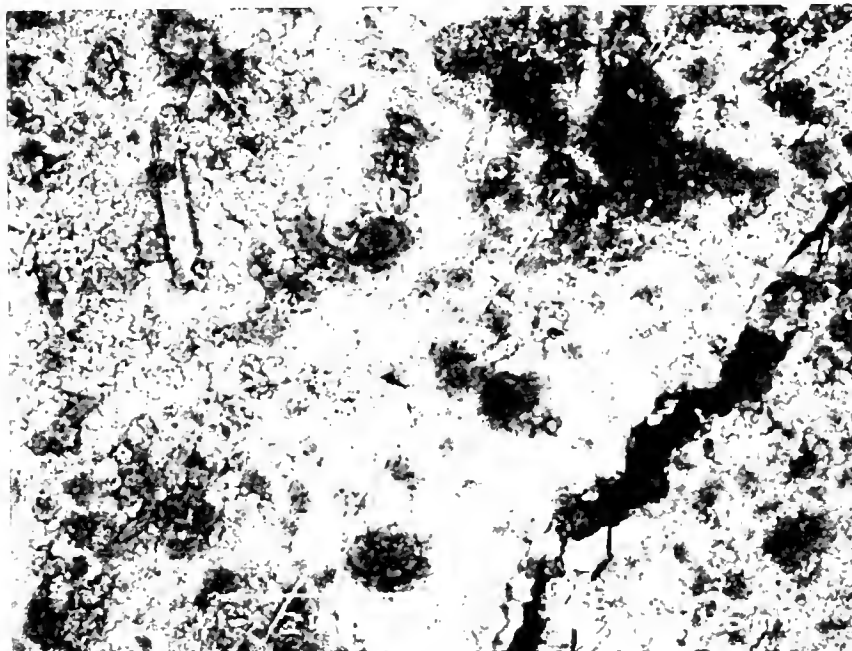
**Magnification:** 125X

**Reflected light**





**Figure 75: Secondary Electron Image of Sample 1: Washroom of Mrs.  
Lockwood's Room**



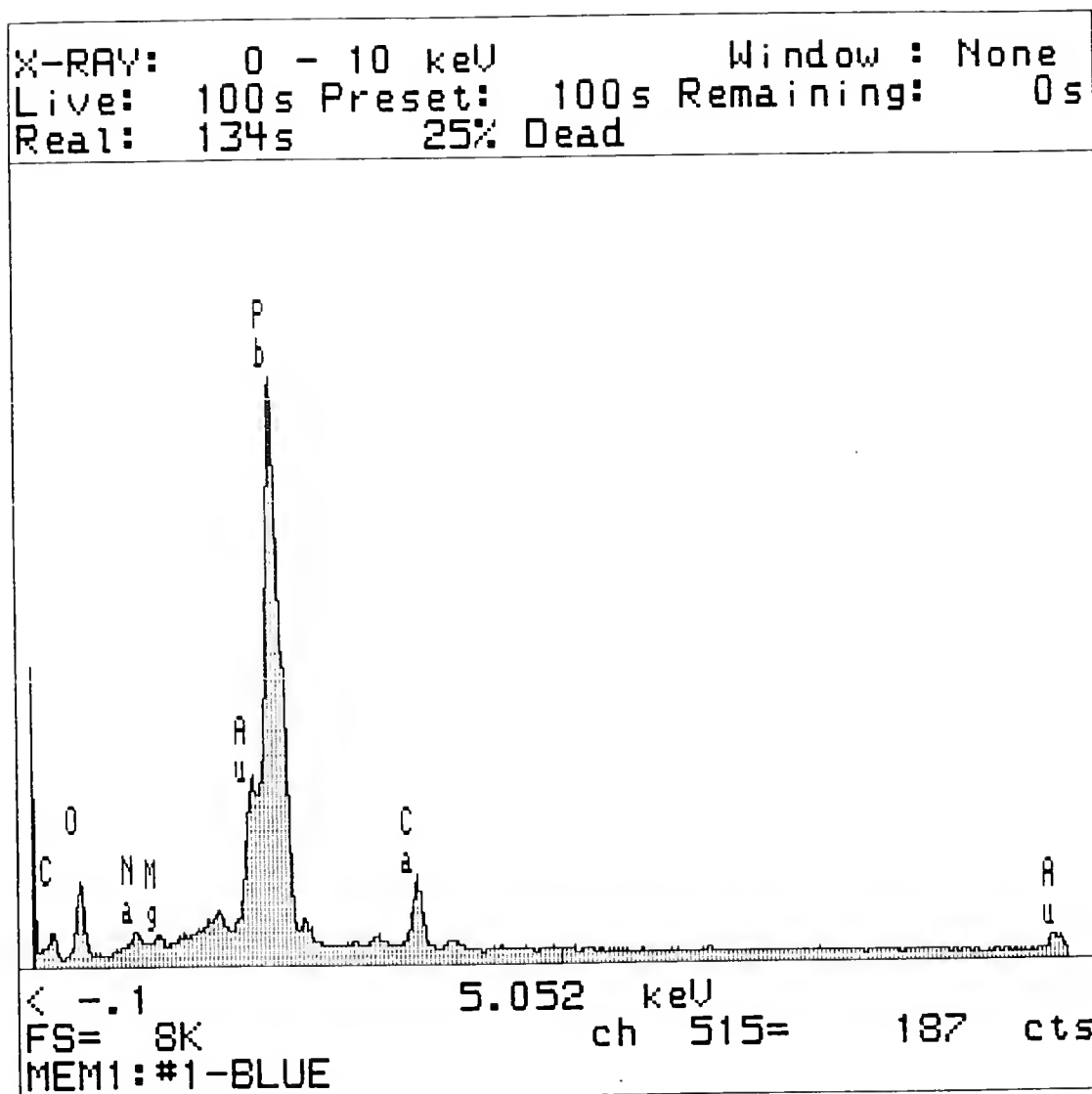
**Figure 76: Back Scatter Electron Image of Sample 1: Washroom of Mrs.  
Lockwood's Room**





**Figure 77: X-Ray Energy Dispersive Analysis of Sample 1: Washroom of Mrs. Lockwood's Room**

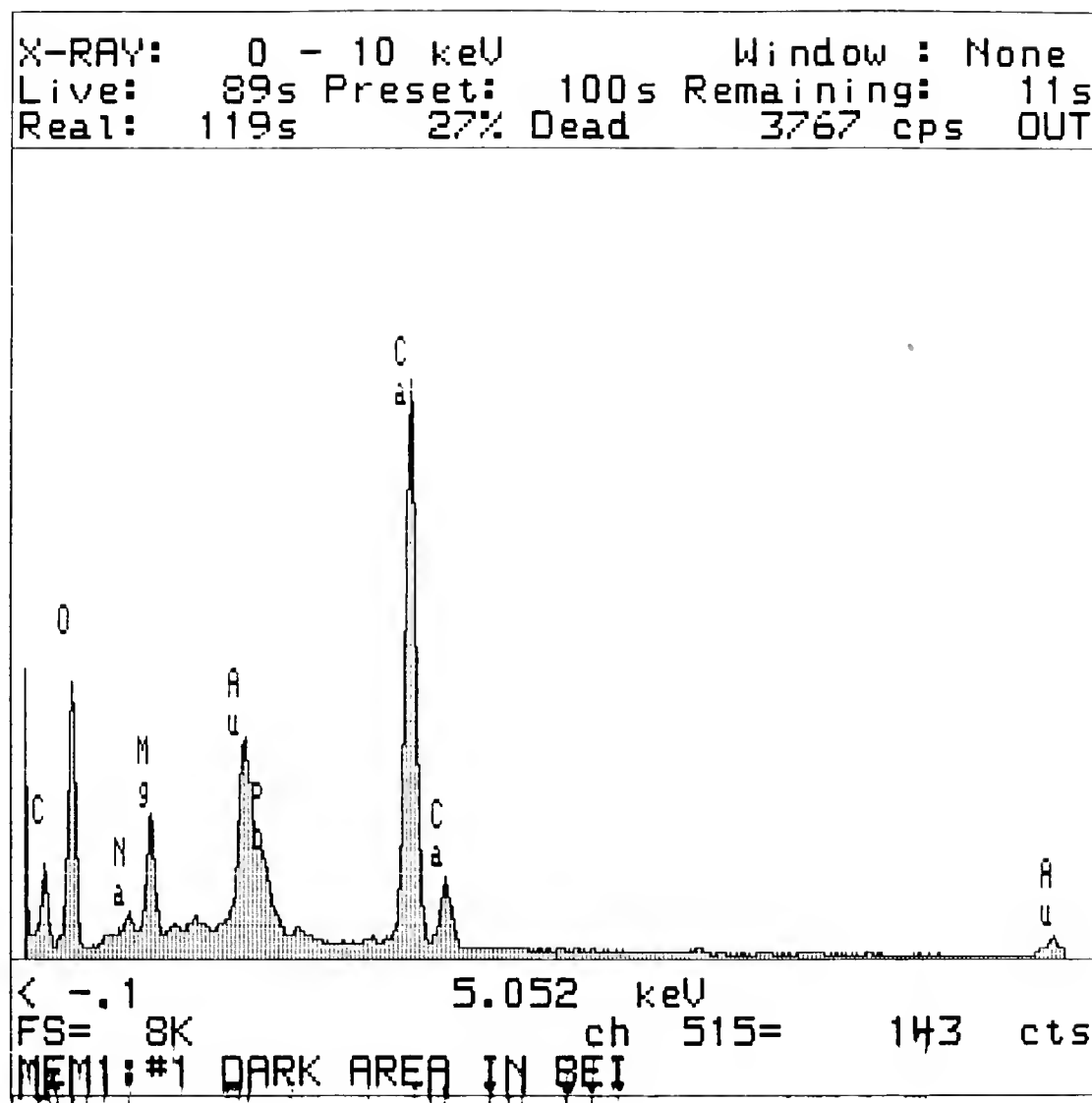
Sample location is just below the cornice. Sample 1 is discussed in Figure 23. The pigment analysis indicated presence of lead and tested positive for ultramarine. This gold coated sample is suggestive of ultramarine and lead oxides.





**Figure 78: X-Ray Energy Dispersive Analysis of Sample 1: Washroom of Mrs. Lockwood's Room**

This is the dark area on the BEI (Fig.76). This gold coated sample is suggestive of ultramarine and lead oxides.







**Figure 79: Photo Micrograph of Sample 3: Washroom of Mrs. Lockwood's Room**

**Sample Location:** Ceiling of Washroom

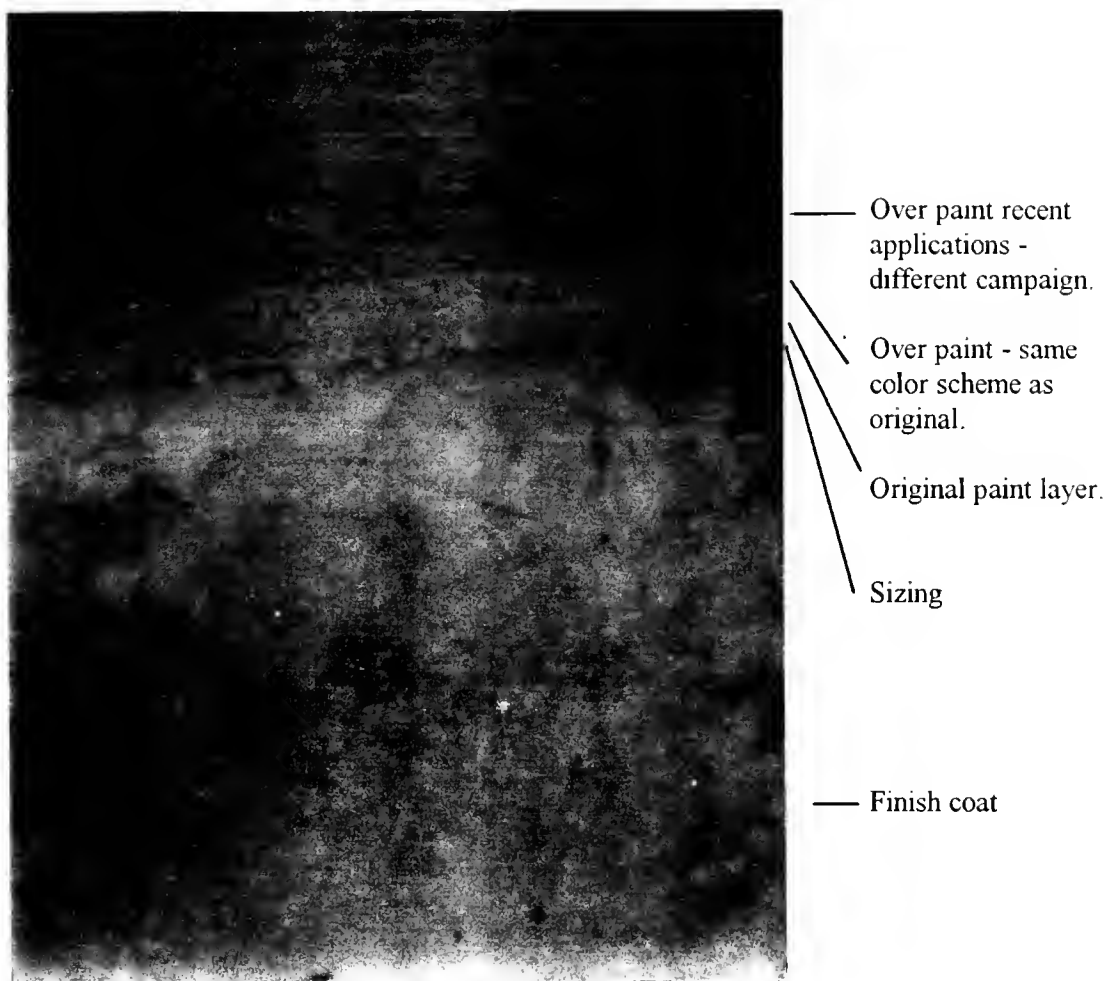
**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 3

**Camera:** Nikon

**Magnification:** 225X

**Reflected light**

**Comments:** Chemical analysis of the green campaigns indicate no lead, but the presence of copper could be verdigris.





**Figure 80: Photo Micrograph of Sample 4A: Washroom of Mrs. Lockwood's Room**

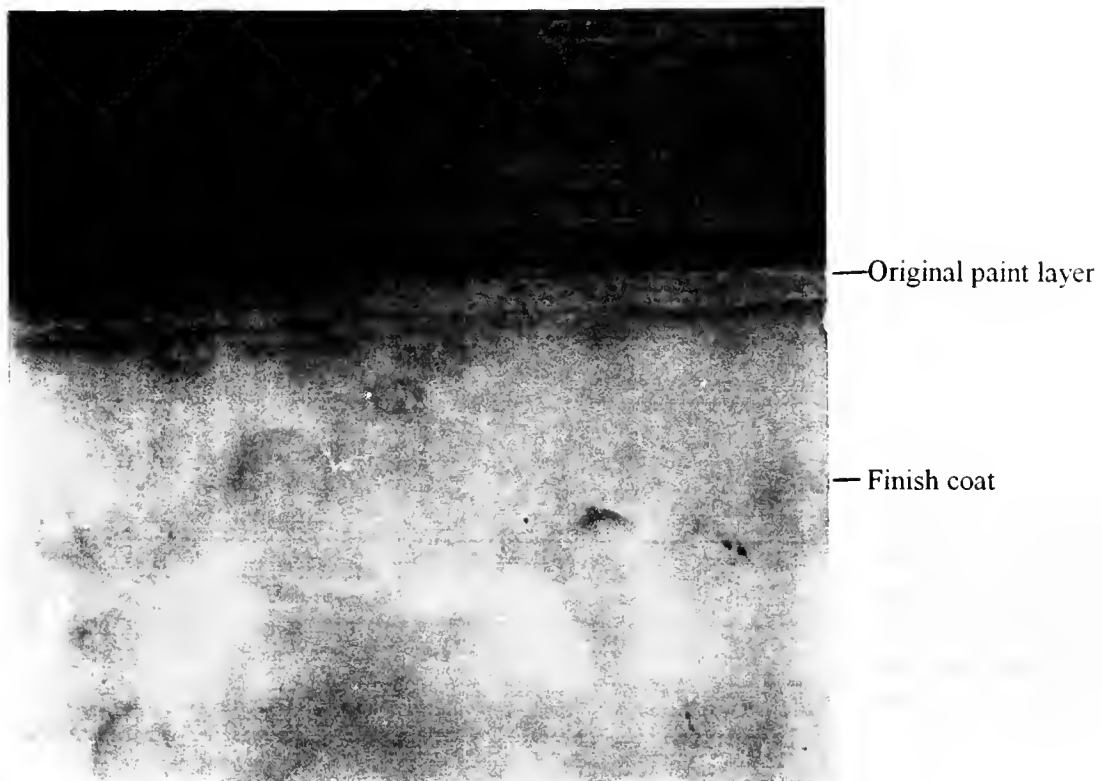
**Sample Location:** Cornice of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 5

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 81: Photograph of Sample 5 Area: Washroom of Mrs. Lockwood's Room**

**Sample Location:** Wall of Washroom

**Camera:** Minolta

Sample 5A-C



Sample 5 was taken from part of the field, the red border, the black line, and the very light grey inside of the border.



**Figure 82: Photo Micrograph of Sample 5A: Washroom of Mrs. Lockwood's Room**

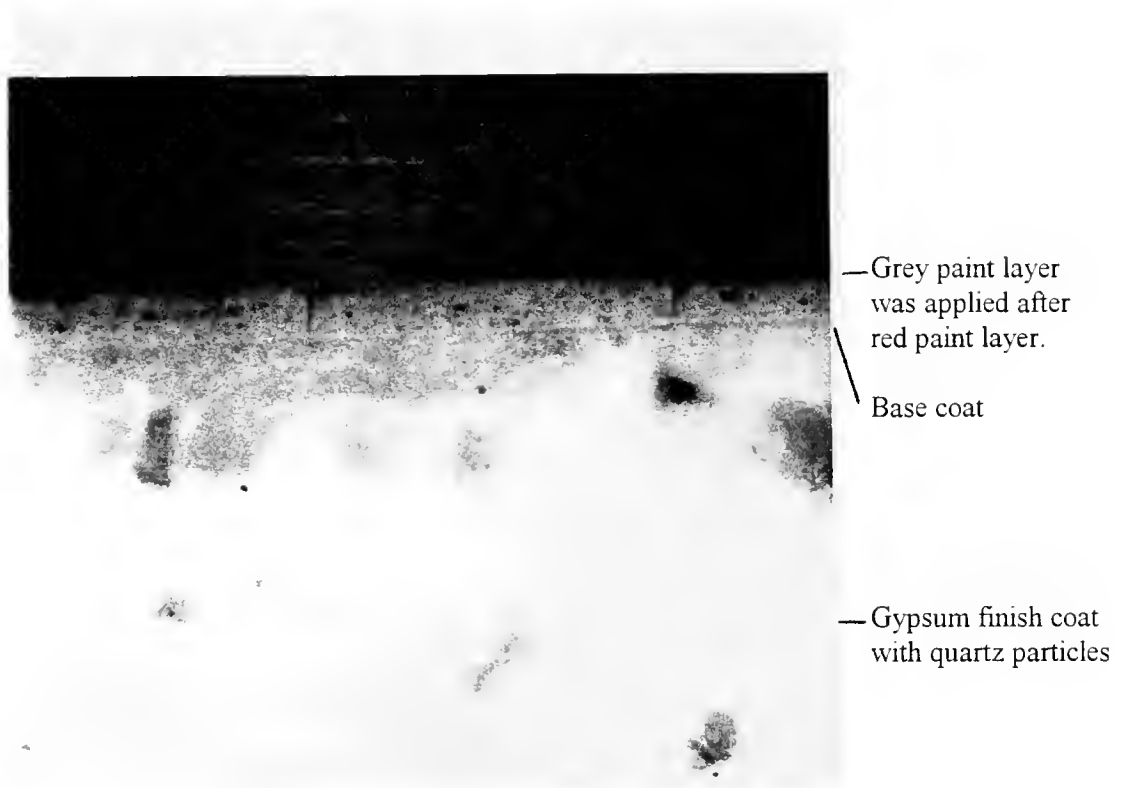
**Sample Location:** Cornice of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 7

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**







**Figure 83: Photo Micrograph of Sample 5B: Washroom of Mrs. Lockwood's Room**

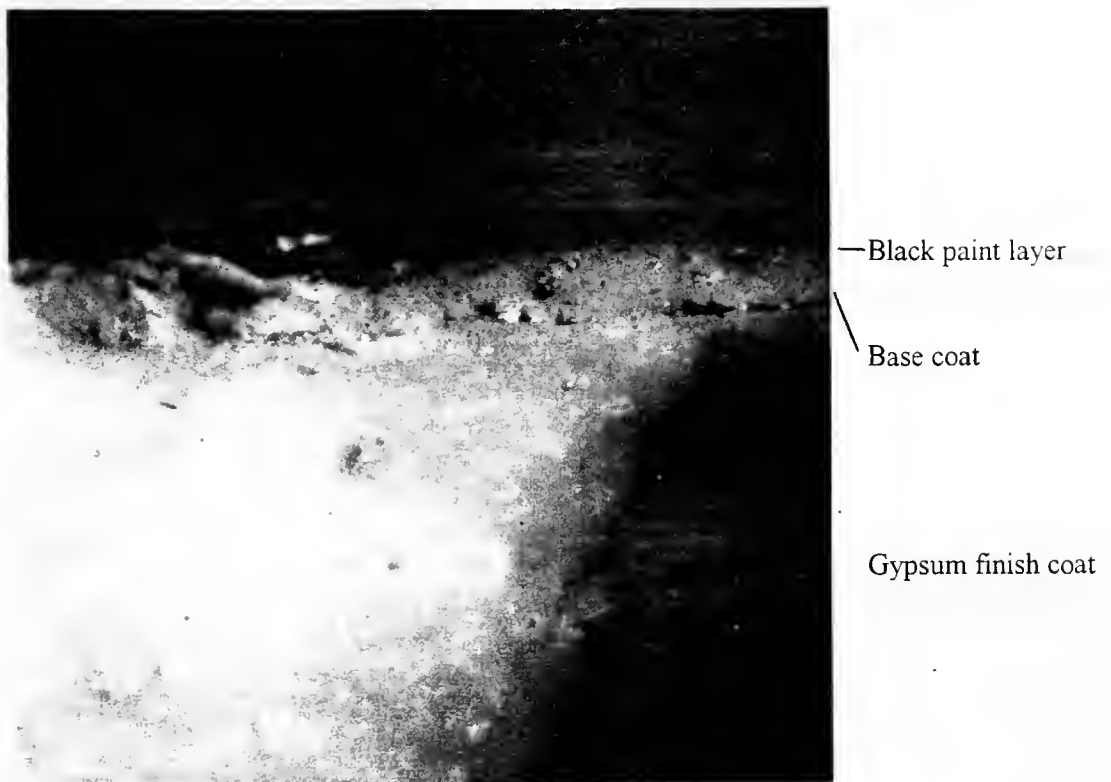
**Sample Location:** Cornice of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 8

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 84: Photo Micrograph of Sample 5C: Washroom of Mrs. Lockwood's Room**

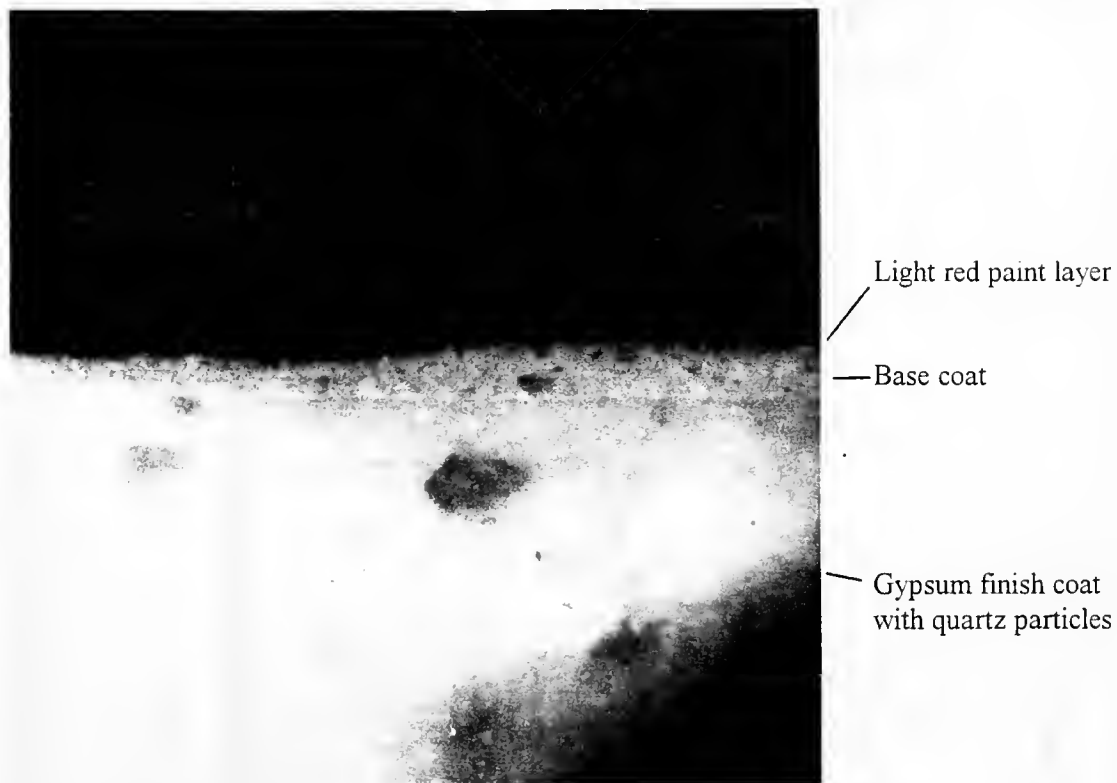
**Sample Location:** Cornice of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 9

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 85: X-Ray Energy Dispersive Analysis of Sample 5: Washroom of Mrs. Lockwood's Room**

This gold coated sample of the black pigment could be carbon black, iron oxides and green earth.

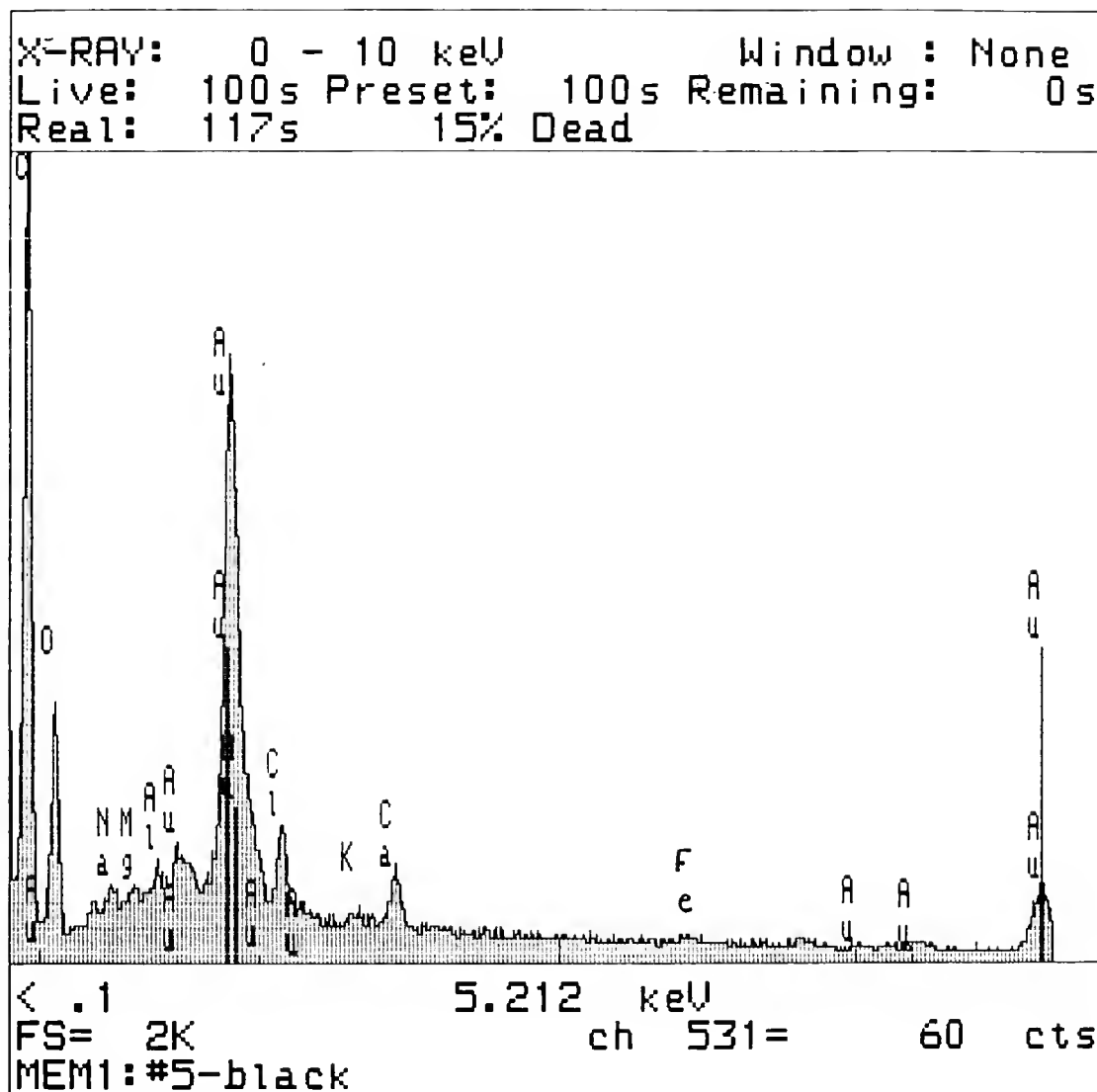
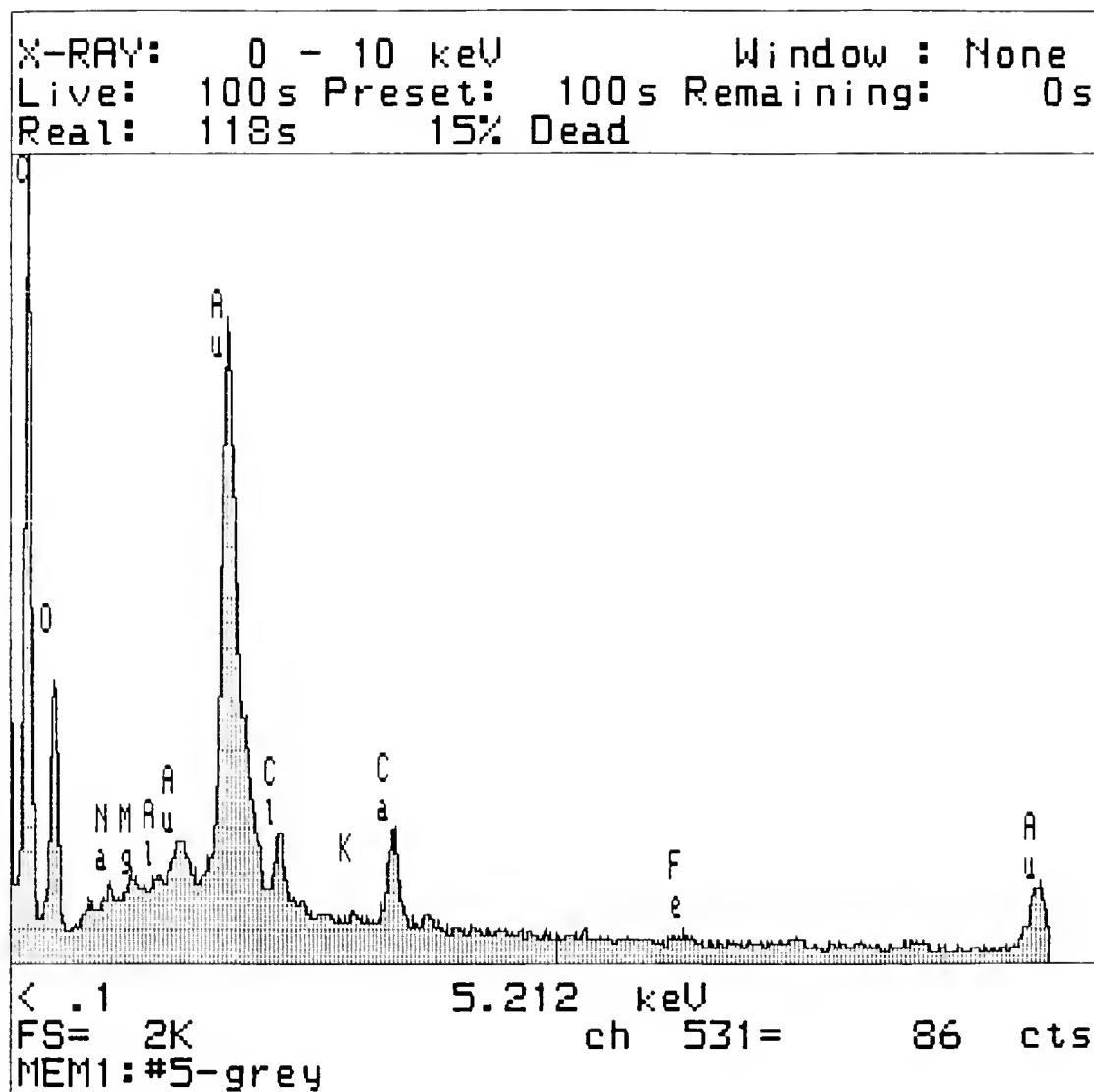




Figure 86: X-Ray Energy Dispersive Analysis of Sample 5: Washroom of Mrs. Lockwood's Room

This gold coated sample of the grey painted area seems to be similar to figure 88; carbon black, iron oxides and green earth again but more whiting in the sample.

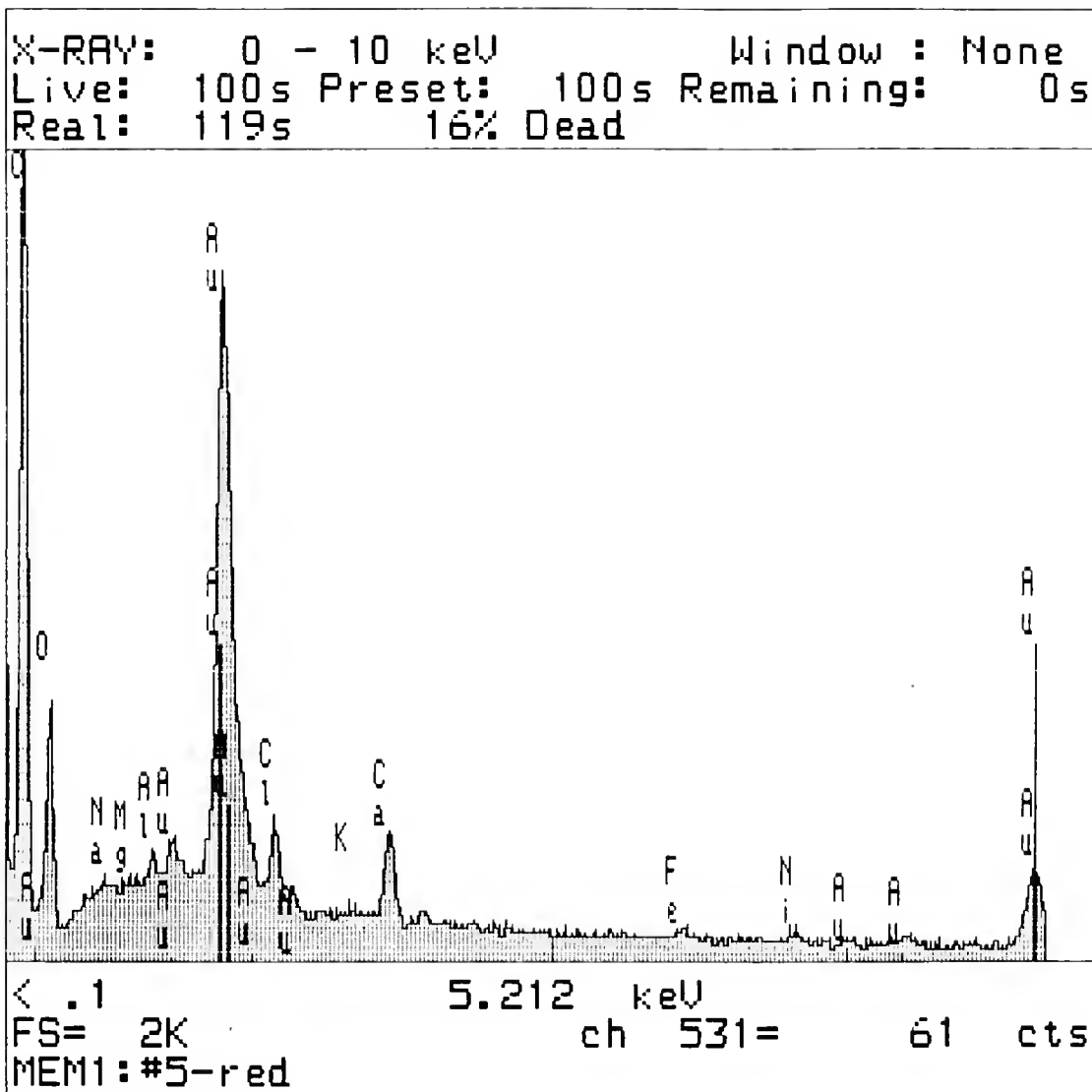






**Figure 87: X-Ray Energy Dispersive Analysis of Sample 5: Washroom of Mrs. Lockwood's Room**

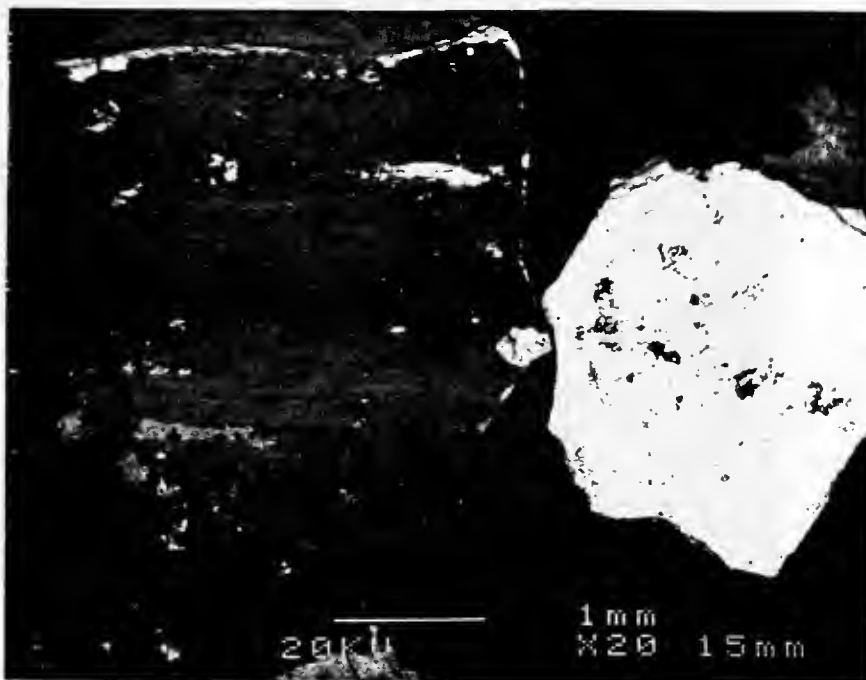
This gold coated sample of the red painted area seems to have alizarin crimson or ultramarine red. The nickel and chlorine which indicates organic pigments cannot be accounted.





**Figure 88: Back Scatter Electron Image of Sample 5 and 37: Washroom of Mrs. Lockwood's Room and Moorish Room.**

This gold coated sample is a comparison between two black painted areas in the rooms. It shows clearly that sample 5 with the black painted area has no lead in it. The black painted area of sample 37 does have lead in it. The dark sample is sample 5 and the light is sample 37.





**Figure 89: Photo Micrograph of Sample 6: Washroom of Mrs. Lockwood's Room**

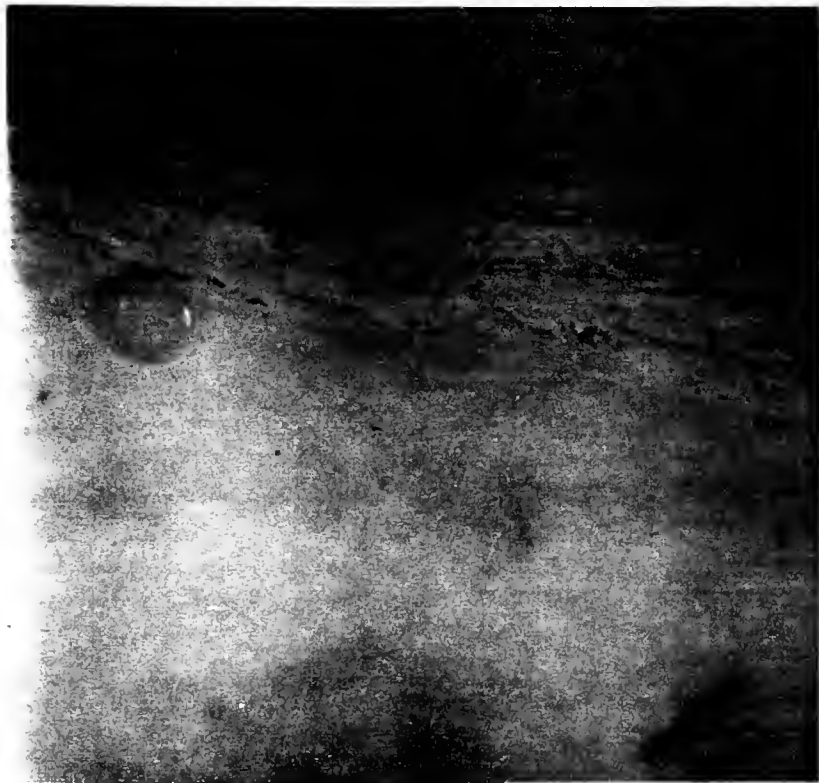
**Sample Location:** Ceiling of Washroom

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 10

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



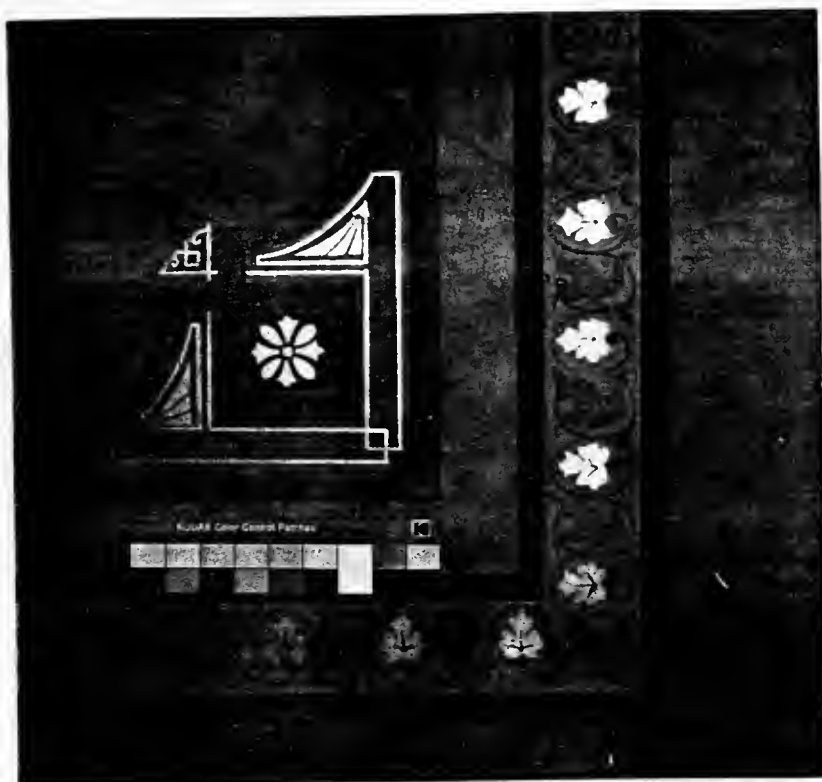
Over paint green  
campaigns looks  
similar to sample 3

Beige campaigns

Gypsum finish coat  
with quartz particles



**Figure 90: Photograph of Sample 9-13 Area: Mrs. Lockwood's Room**







**Figure 91: Photo Micrograph of Sample 9: Wall of Mrs. Lockwood's Room**

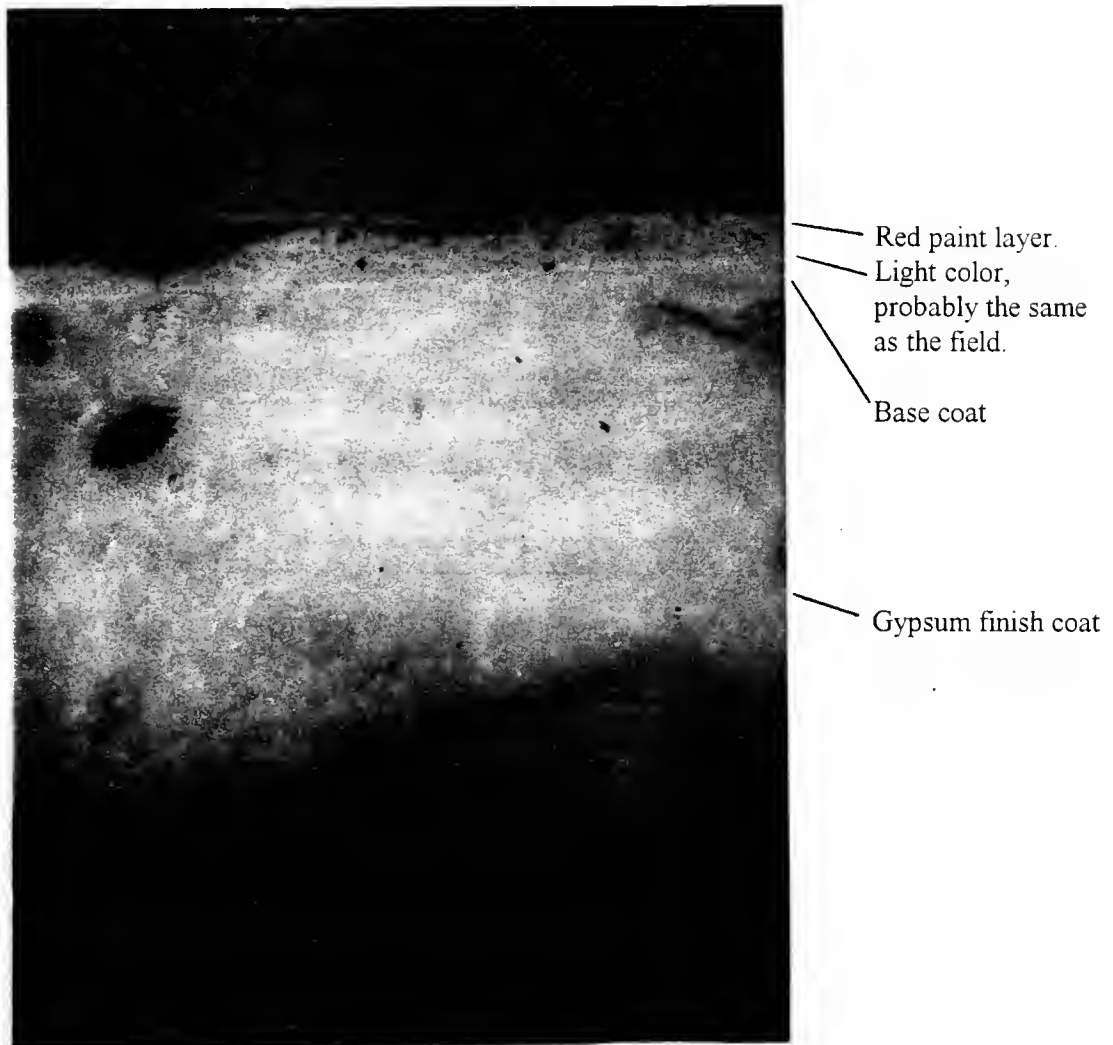
**Sample Location:** East wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 15

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 92: Photo Micrograph of Sample 10A: Wall of Mrs. Lockwood's Room**

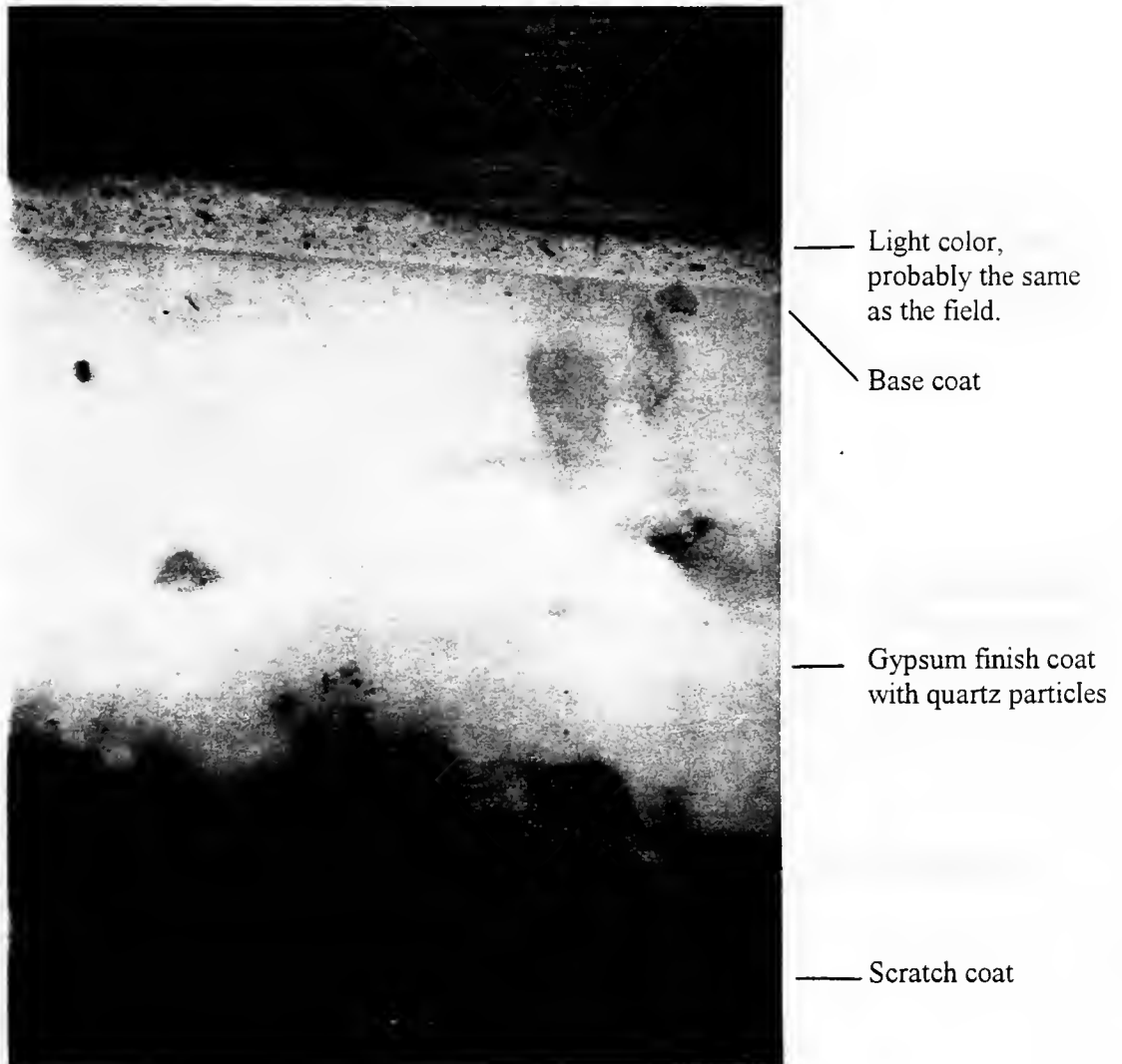
**Sample Location:** East wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 18

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 93: Photo Micrograph of Sample 11: Wall of Mrs. Lockwood's Room**

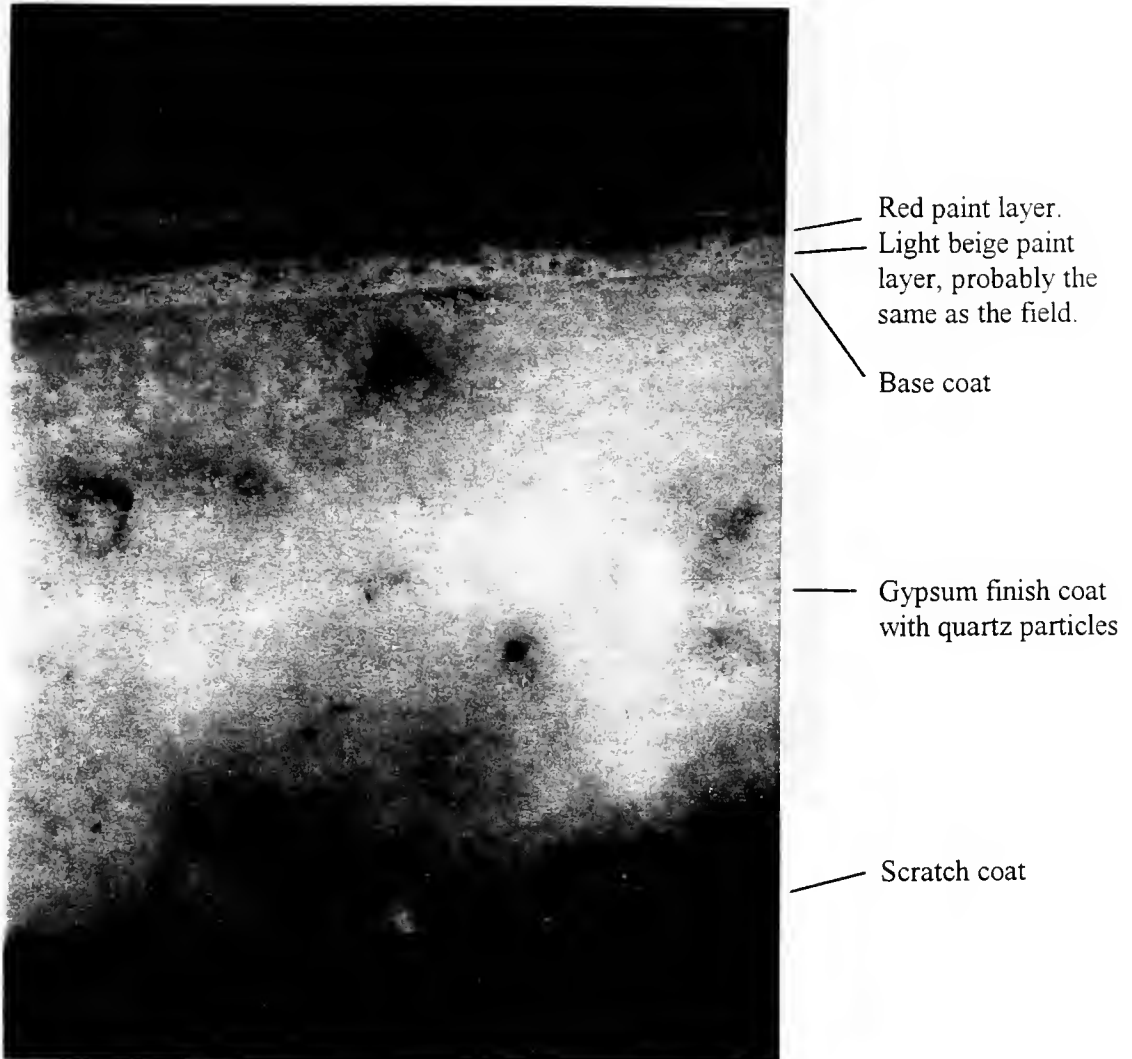
**Sample Location:** East wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 17

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 94: Photo Micrograph of Sample 12: Wall of Mrs. Lockwood's Room**

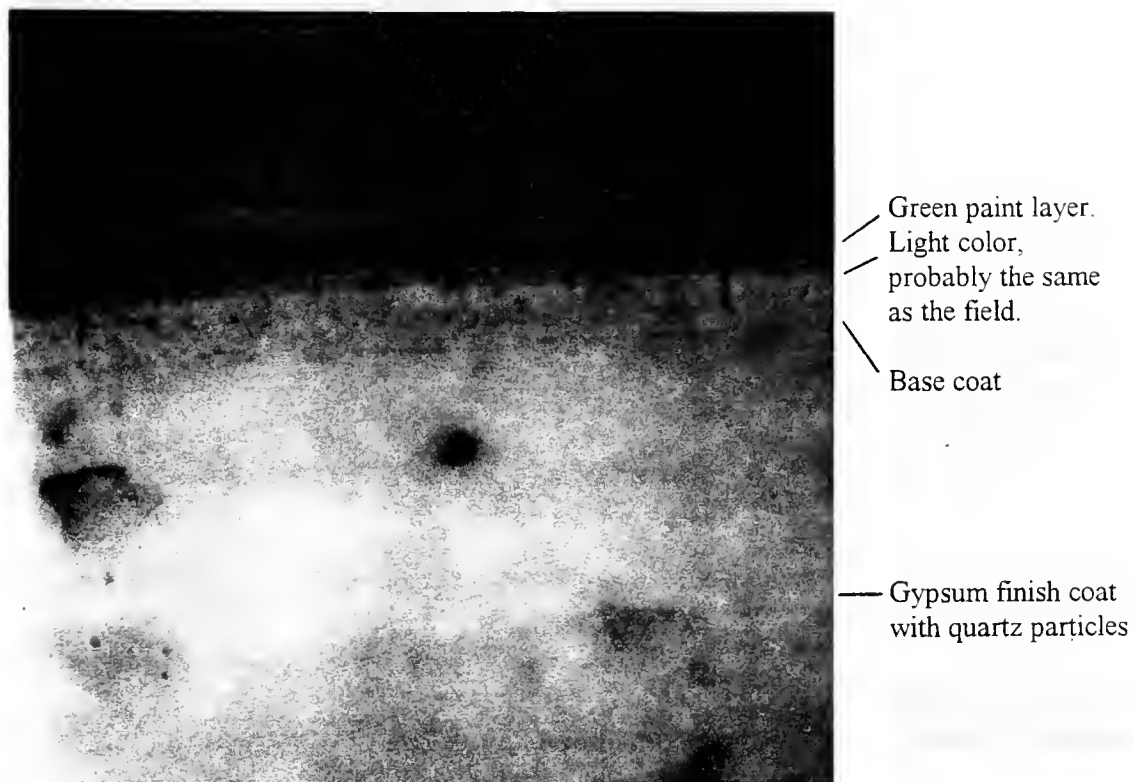
**Sample Location:** East wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 20

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**







**Figure 95: Photo Micrograph of Sample 13: Wall of Mrs. Lockwood's Room**

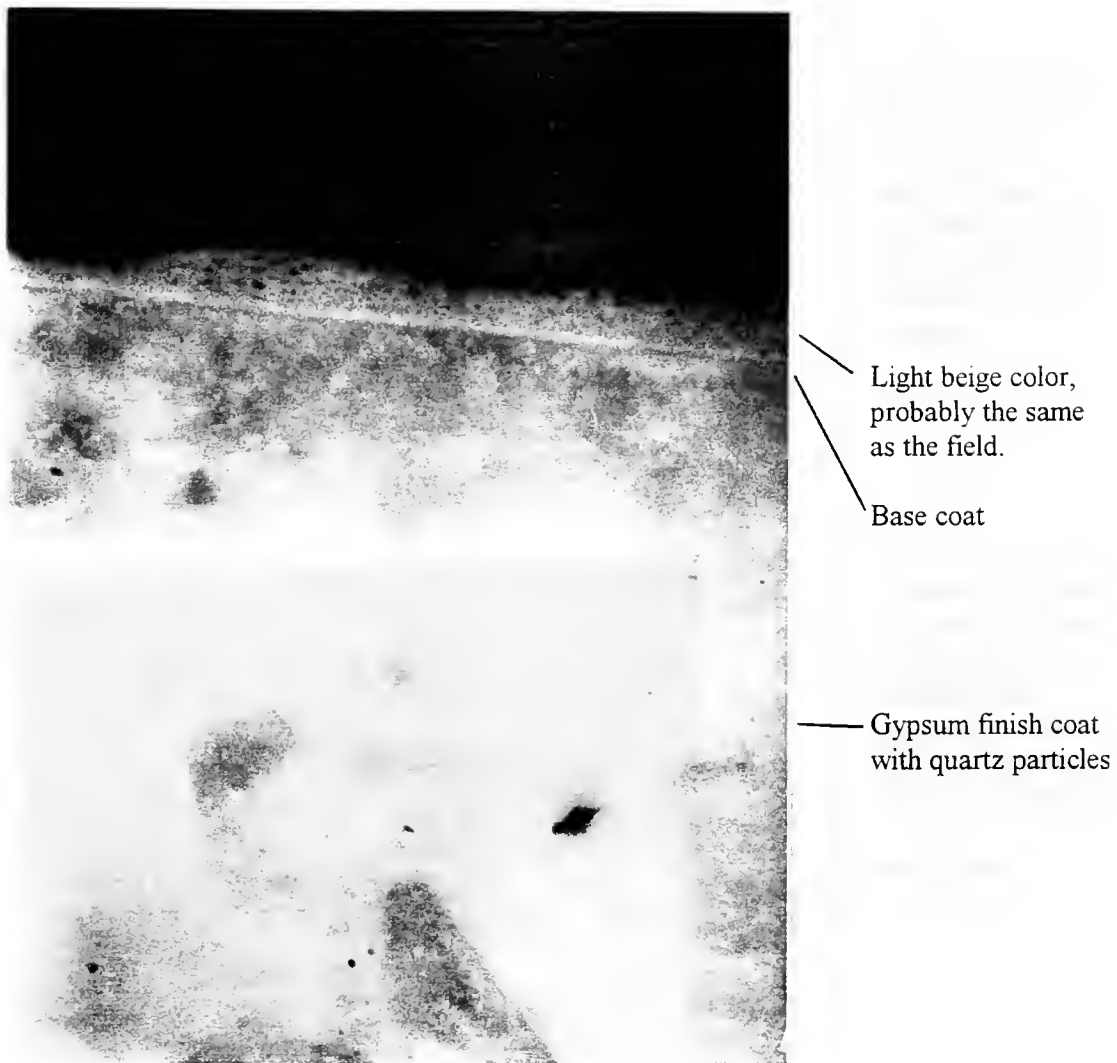
**Sample Location:** East wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 21

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 96: Photo Micrographs of Sample 14: Mrs. Lockwood's Room**

**Sample Location:** North wall

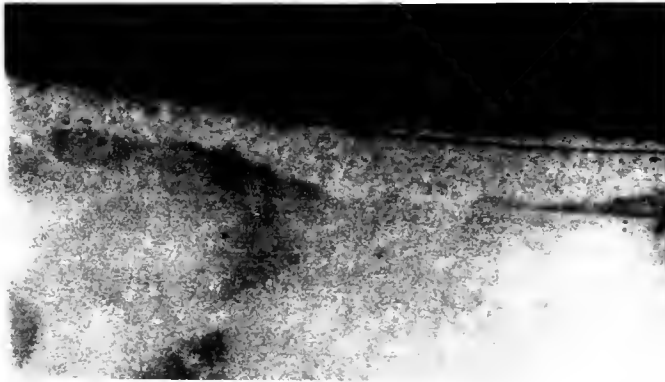
**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 23 and 25

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

Painted area under electrical outlet.



Original paint layer  
with one thin paint  
layer on top  
indicating that  
repainting was done  
shortly before this  
electrical outlet was  
mounted.

Finish coat

Painted area exposed.



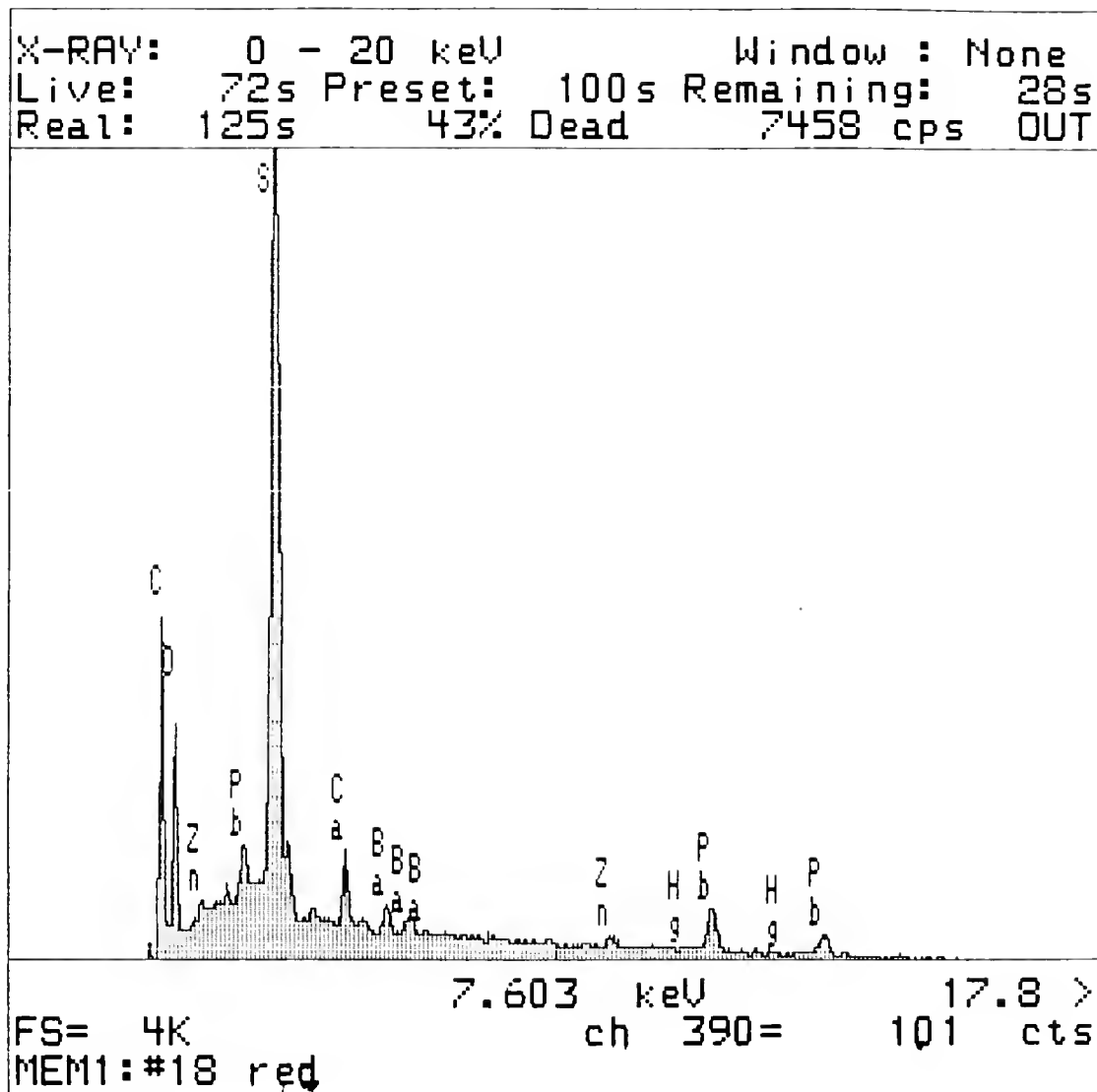
Original paint layer  
with two thin paint  
layers on top,  
indicating that  
repainting was done  
after the electrical  
outlet was mounted.

Finish coat



Figure 97: X-Ray Energy Dispersive Analysis of Sample 18: Mrs Lockwood's Rm

This sample is the red border on the wall. Lithopone is present. No mercury was found which could have explained the red color. This pigment might be alizarin crimson.





**Figure 98: Photo Micrographs of Sample 19A and B: Mrs. Lockwood's Room**

**Sample Location:** Ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 18 and 19

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

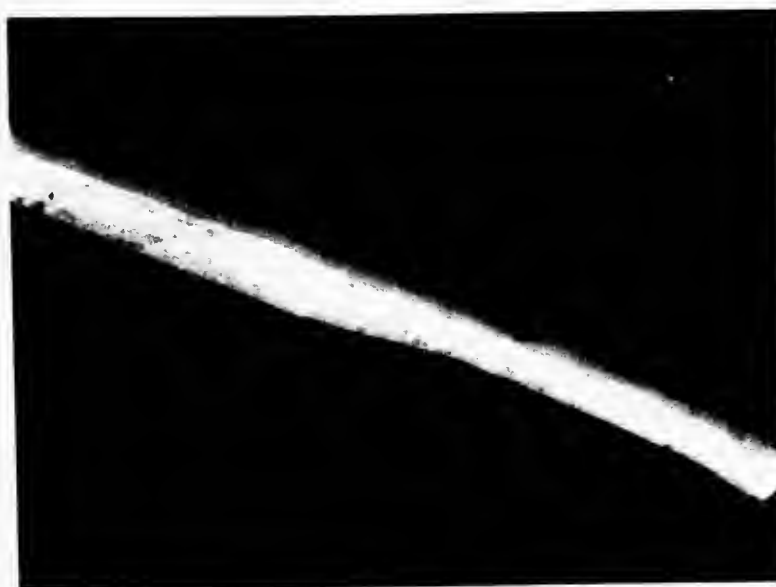
Green paint



Green paint and  
gold layer

Base coat, no  
substrate

Grey paint



Grey paint layer

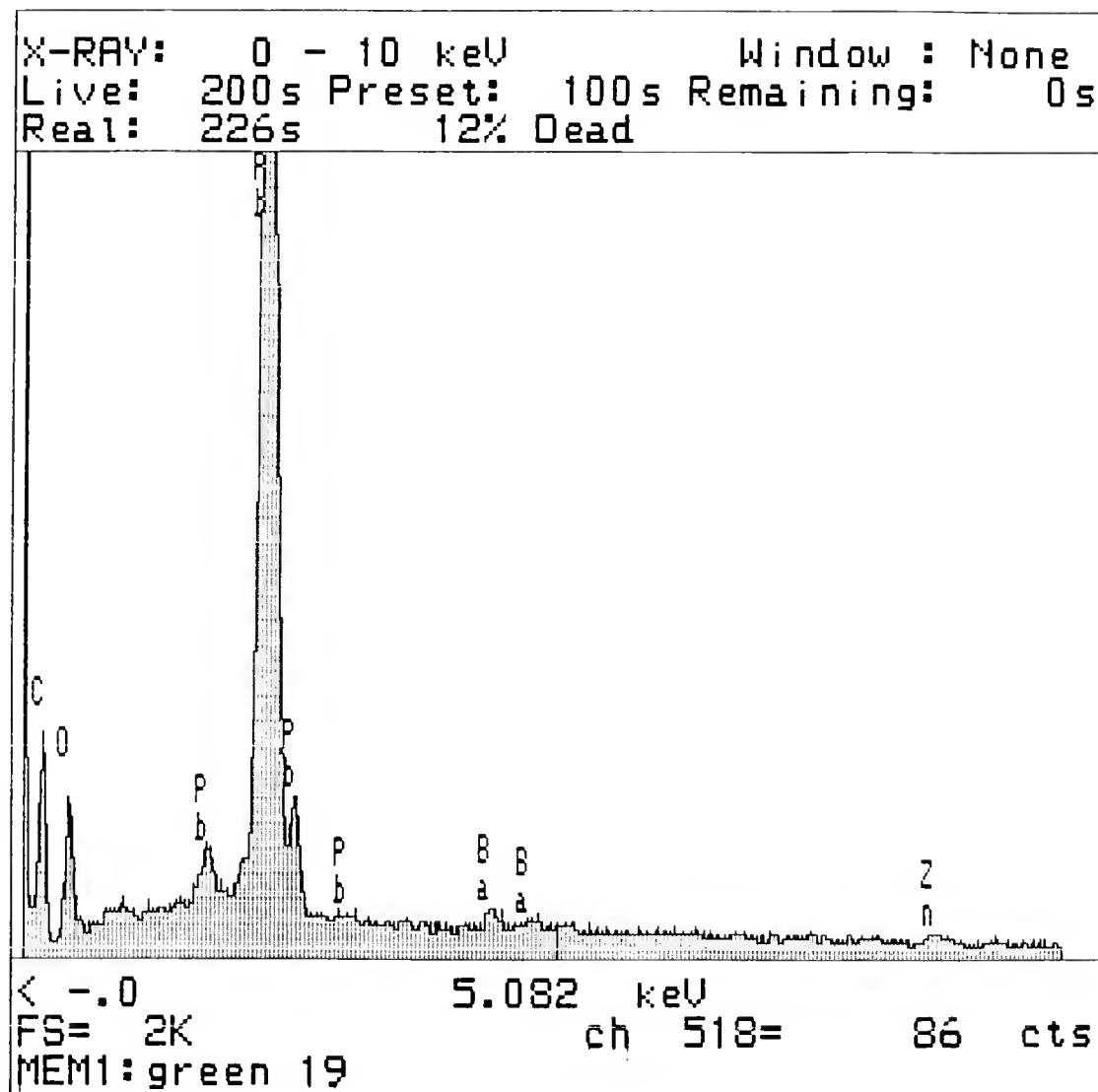
Base coat, no  
substrate





Figure 99: X-Ray Energy Dispersive Analyses of Sample 19: Mrs. Lockwood's Rm

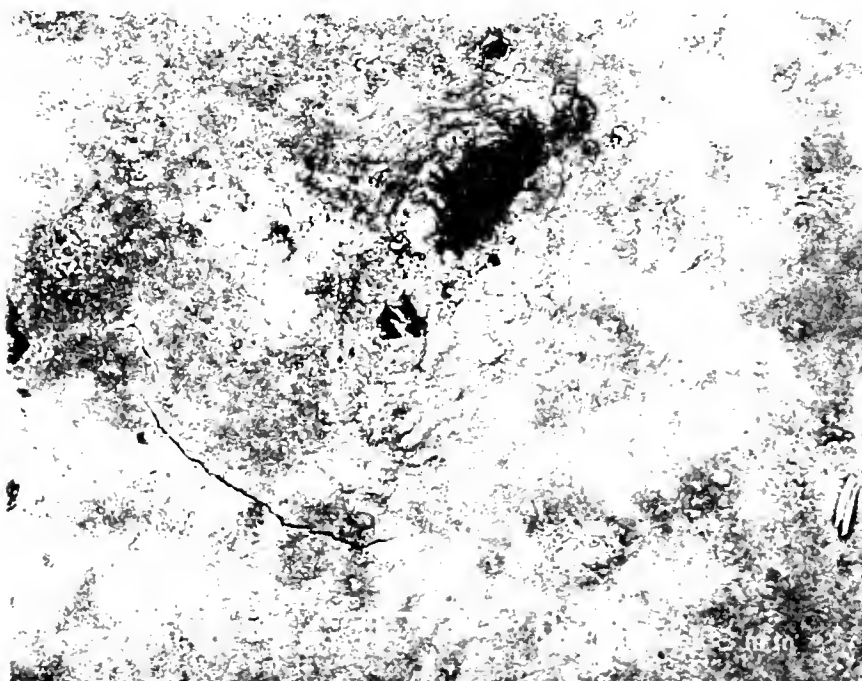
The carbon coated sample of the green painted area reveals lithopone and maybe zinc green.





**Figure 100: X-Ray Energy Dispersive Analysis of Sample 19: Mrs. Lockwood's Rm**

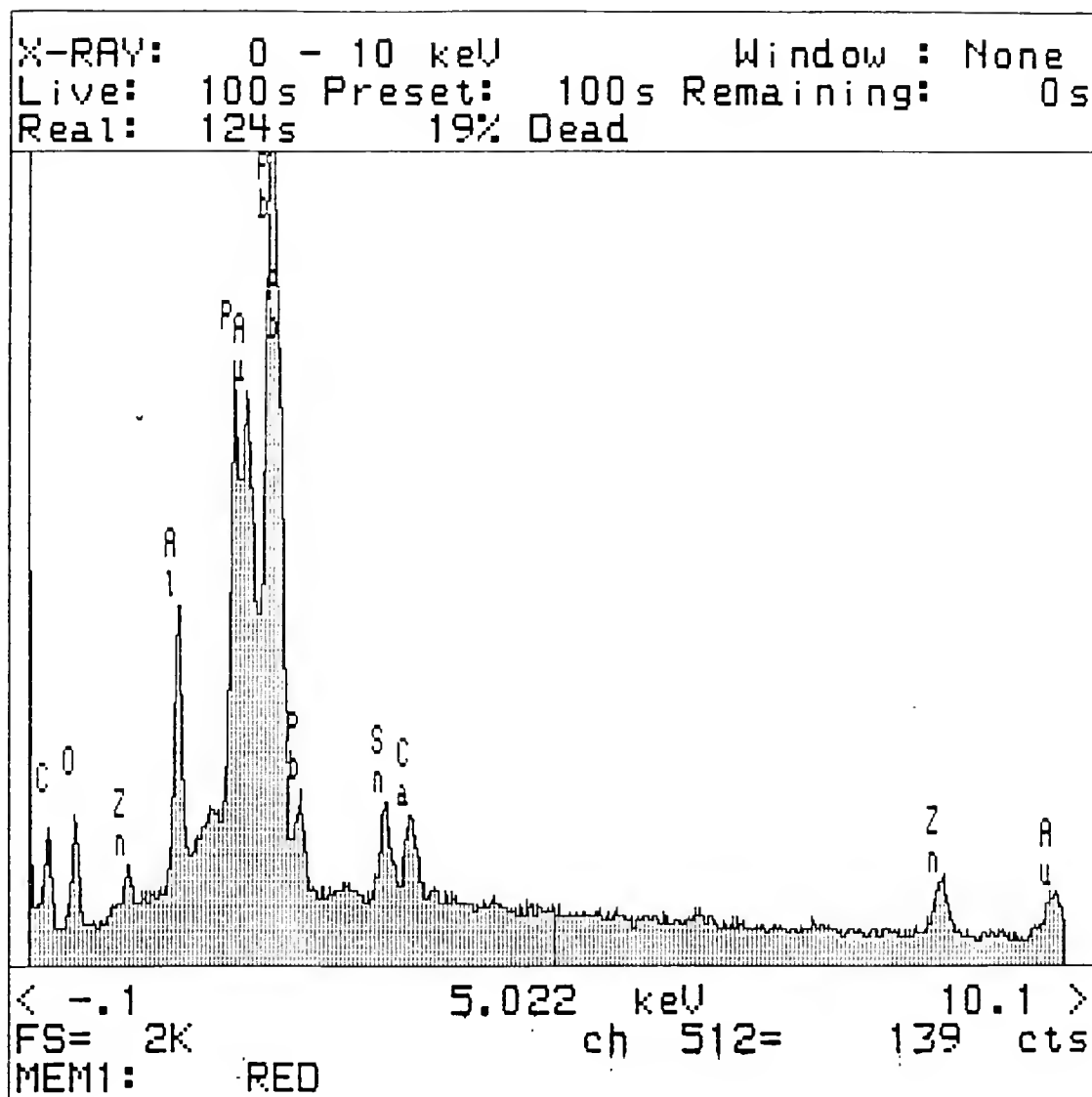
It is possible that this carbon coated sample of a red painted area on the ceiling was made with alizarin crimson.





**Figure 101: Back Scatter Electron Image of Sample 19: Mrs. Lockwood's Room**

This sample is from the ceiling. It is a red painted sample which was gold coated for analysis. It looks like alizarin crimson optically. The presence of Al seems to be the base for this organic paint. Sn is inconclusive.





**Figure 102: Photo Micrographs of Sample 20 A-B: Mrs. Lockwood's Room**

**Sample Location:** Ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 21, Film 2 Negative 22

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

Sample 20A: Grey paint



Sample 20 B: Red paint







Figure 103: X-Ray Energy Dispersive Analysis - Sample 21: Mrs. Lockwood's Rm

This sample is from the ceiling. This carbon coated sample seems to contain lithopone and iron oxide. The beige color is yellow ochre.

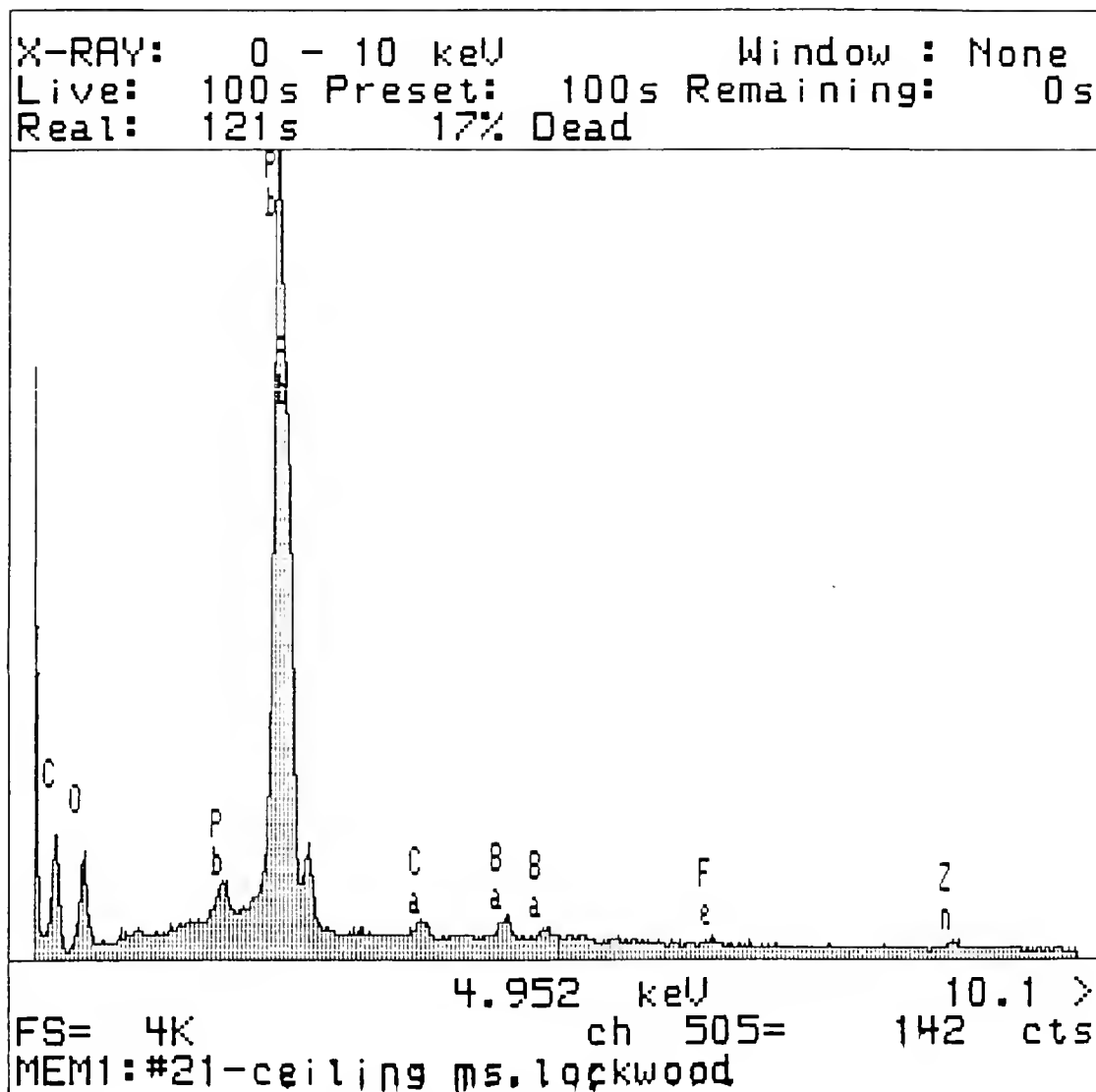




Figure 104: X-Ray Energy Dispersive Analysis - Sample 21: Mrs. Lockwood's Rm

This sample was carbon coated. The sample consists of recent retouch paint which explains the titanium. It is also very high in organic pigments. Also, there is the presence of ultramarine, phthalocyanine, titanium white, and whiting.

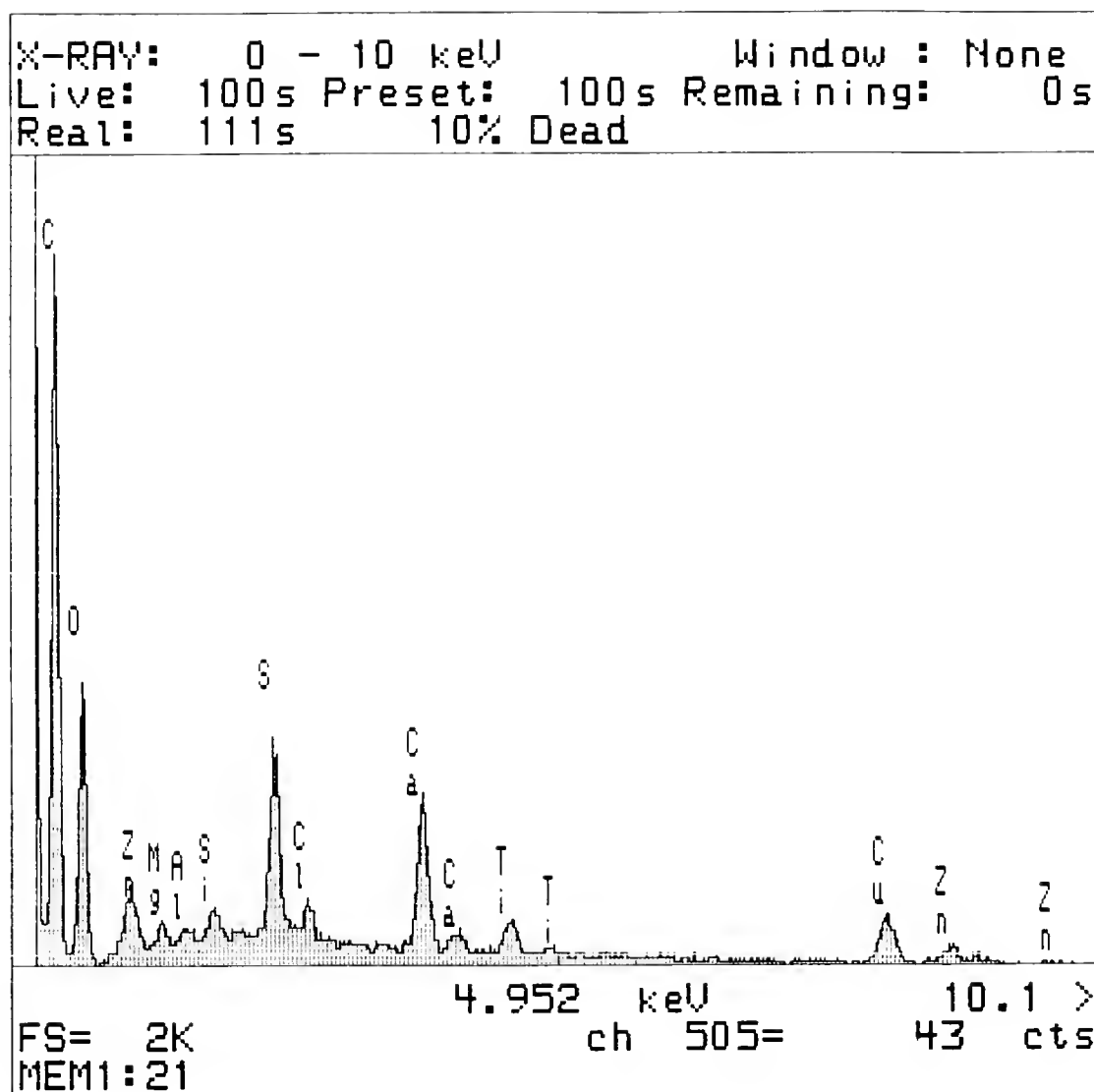




Figure 105: X-Ray Energy Dispersive Analysis - Sample 21: Mrs. Lockwood's Room

This gold coated Sample 21 has no lead. It has a high quantity of titanium, sulphur and calcium carbonate. It contains lithopone and chromium oxide.

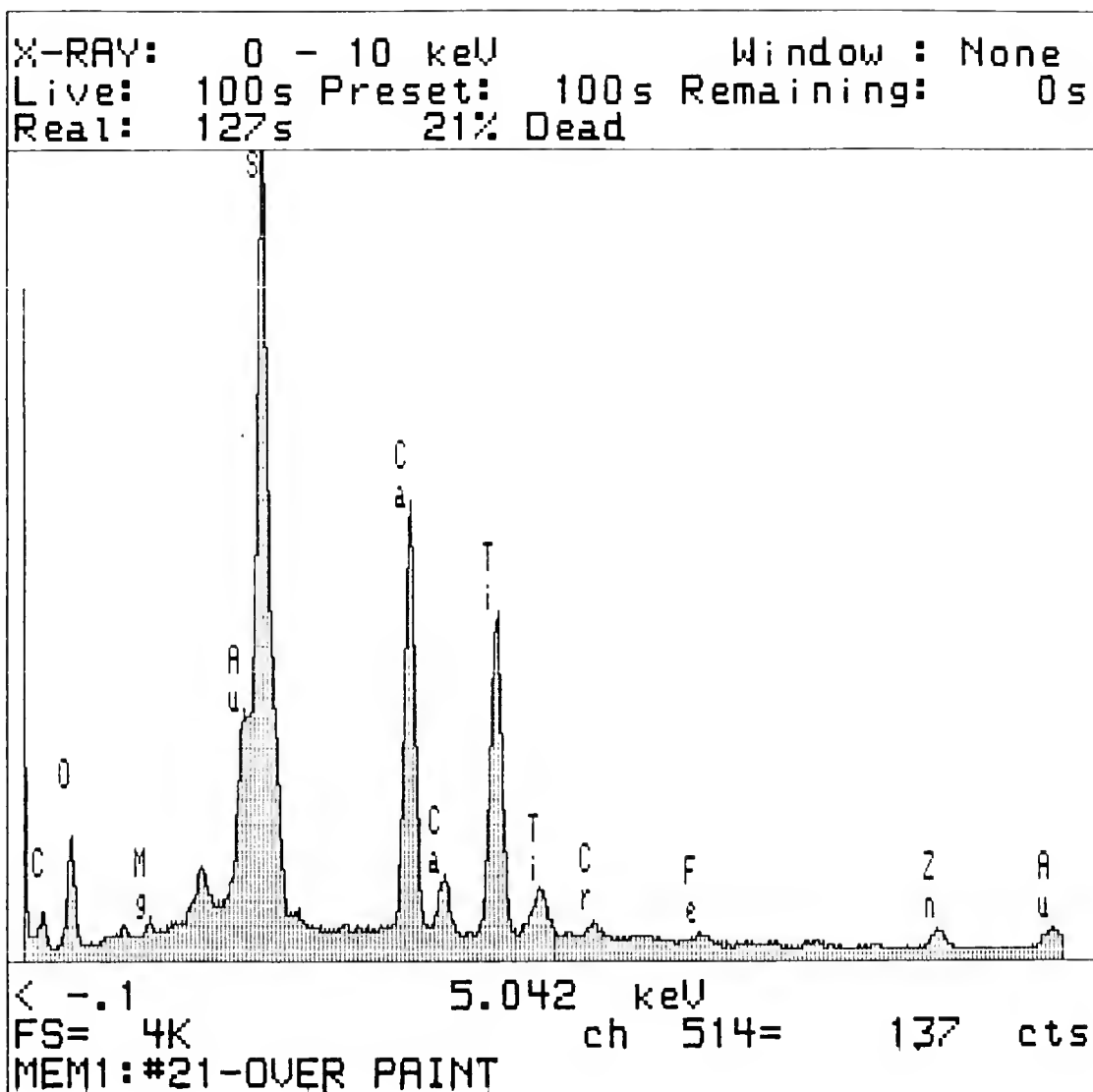
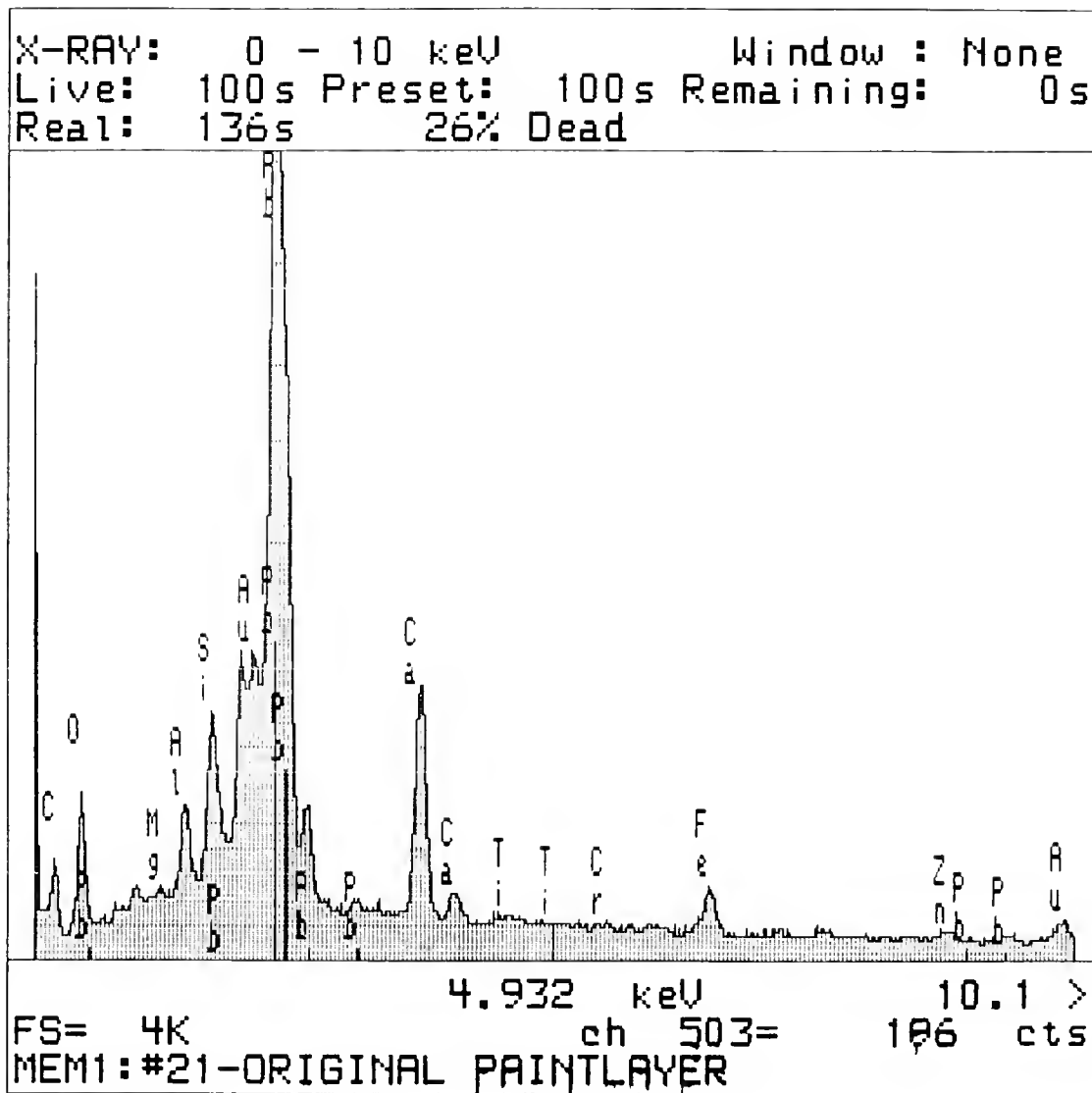




Figure 106: X-Ray Energy Dispersive Analysis - Sample 21: Mrs. Lockwood's Room

This gold coated Sample 21 shows the chemical components of the original paint layer

Lithopone, a green organic pigment, and yellow ochre are present.







**Figure 107: Photo Micrograph of Sample 7: Bathroom of Mrs. Lockwood's Room**

**Sample Location:** Ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 11

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

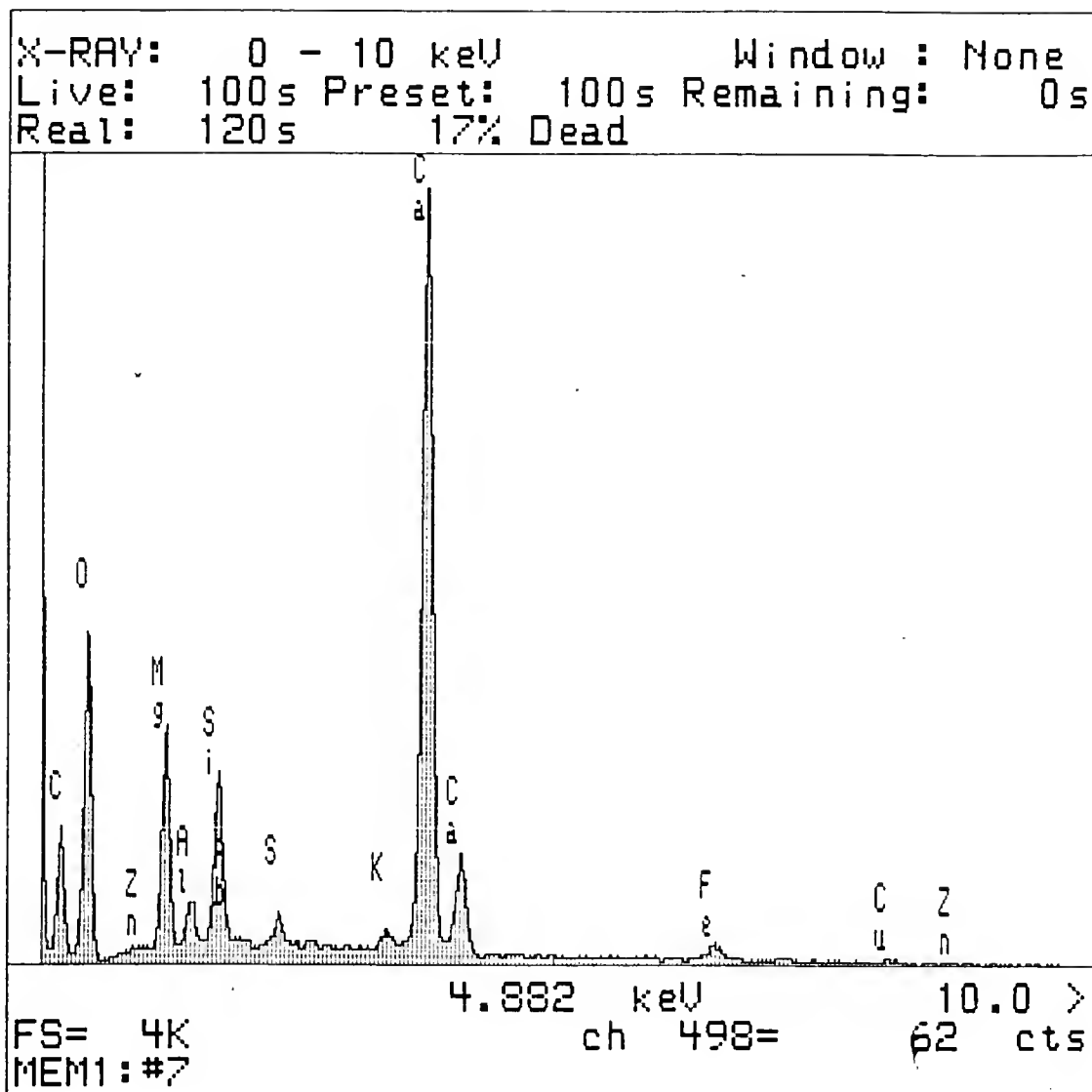


— Red paint layer  
— Base coat  
  
— Finish coat



**Figure 108: X-Ray Energy Dispersive Analysis of Sample: Bathroom of Mrs. Lockwood's Room**

This is a carbon coated sample of the red painted area. It seems that an organic pigment was used such as alizarin crimson. No lead is present. The presence of Cu might be cuprous oxide which was patented in 1867 but was not commonly used at that time.





**Figure 109: Photo Micrograph of Sample 25: Bathroom of Mrs. Lockwood's Room**

**Sample Location:** Ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 30

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



Green campaigns

Red campaigns

Base coat

Finish coat



**Figure 110: Photo Micrograph of Sample 8: Bathroom of Mrs. Lockwood's Room**

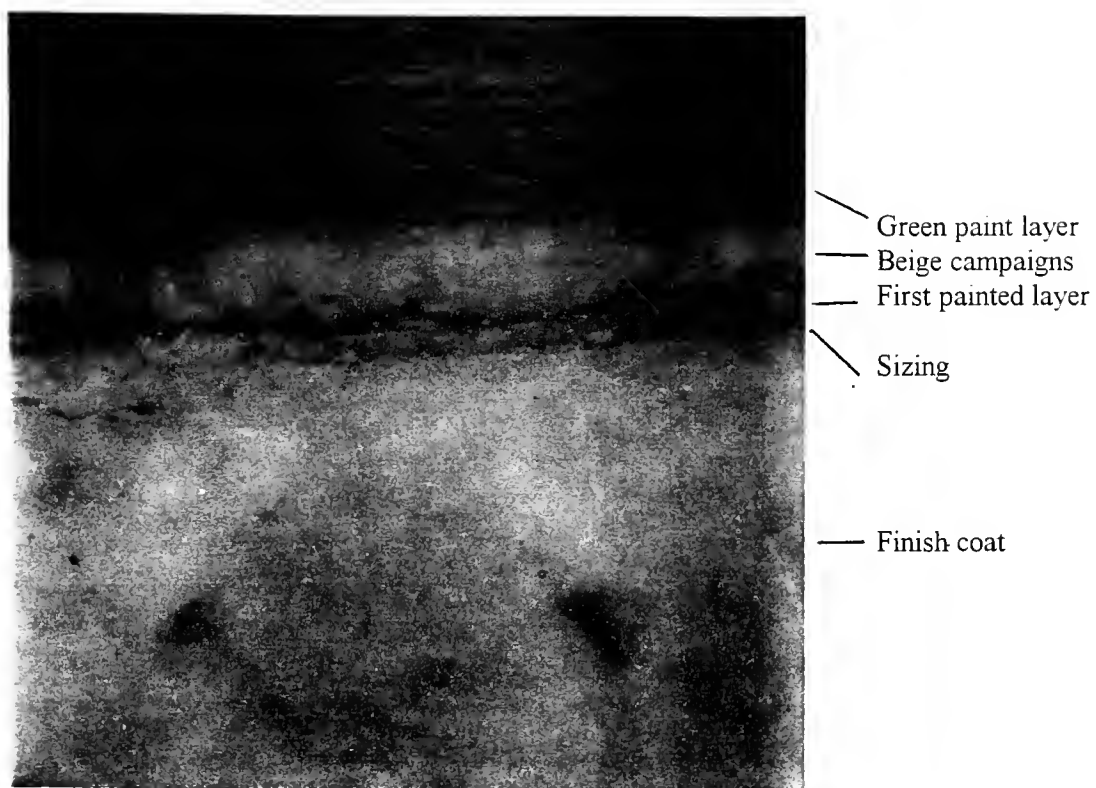
**Sample Location:** Ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 1 Negative 14

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**







**Figure 111: Photo Micrograph of Sample 26: Bathroom of Mrs. Lockwood's Room**

**Sample Location:** Ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 28

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

Similar as Sample 25

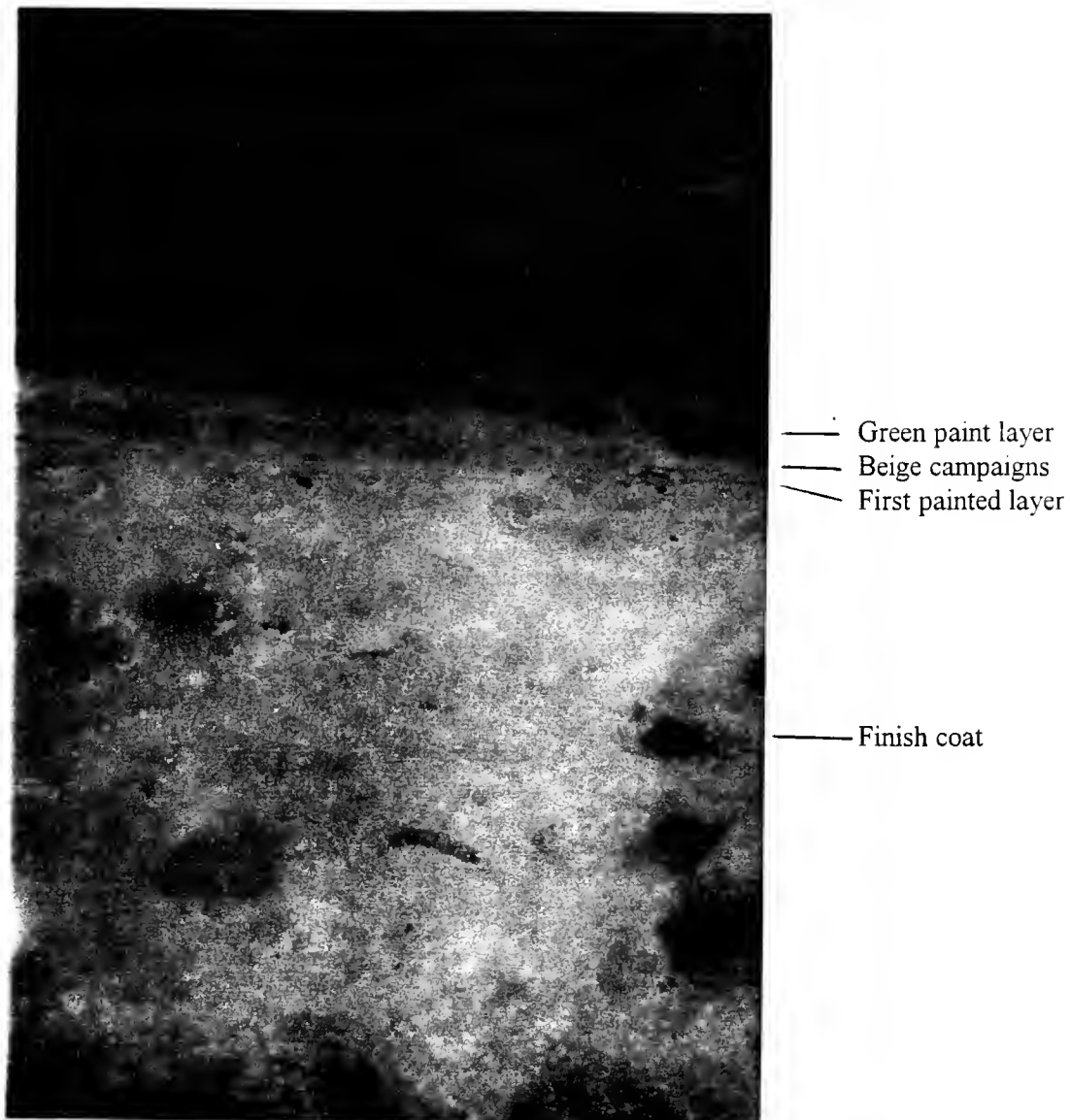




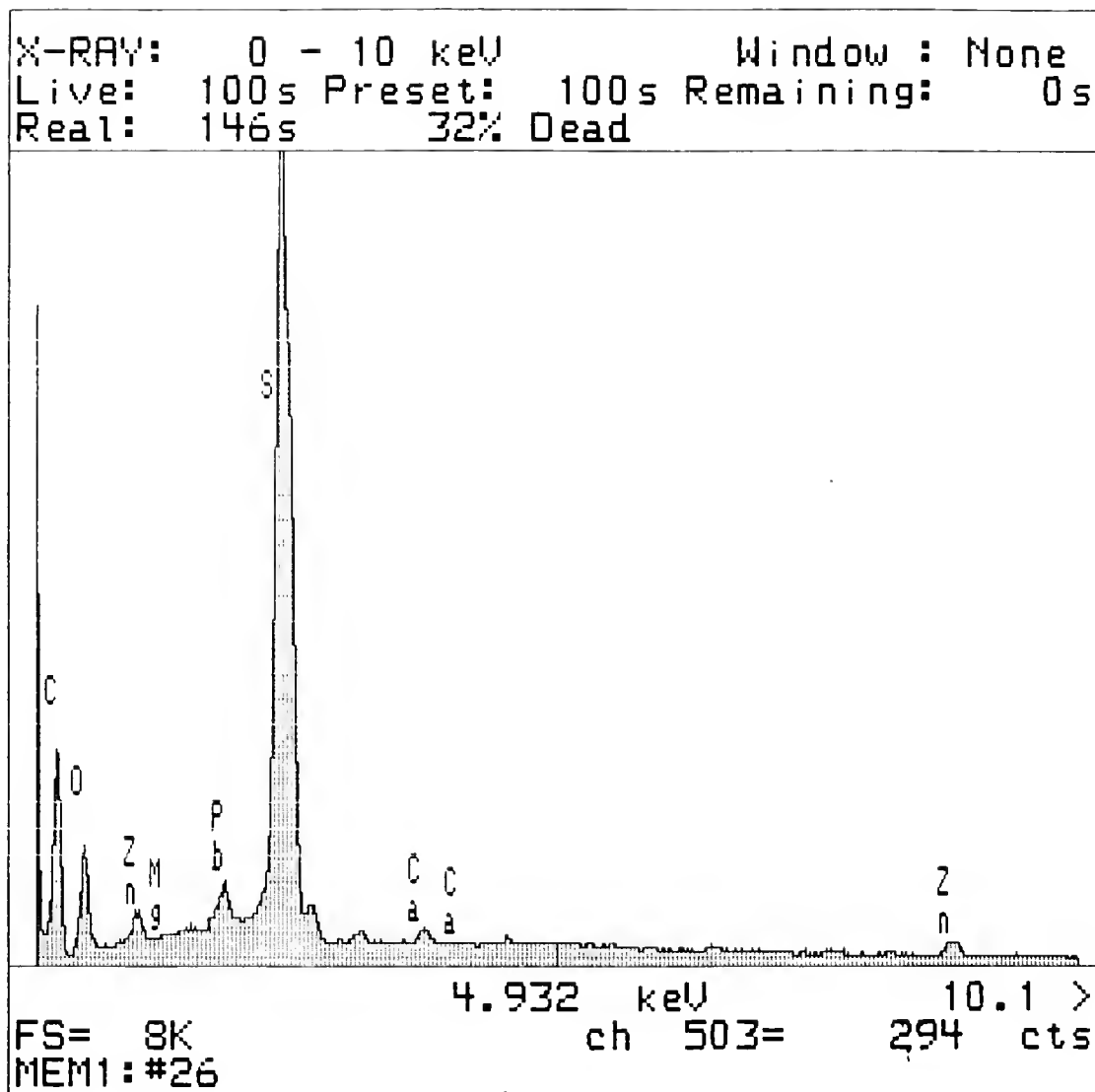
Figure 112: Back Scatter Electron Image of Sample 26: Bathroom of Mrs. Lockwood's Room





**Figure 113: X-Ray Energy Dispersive Analysis of Sample 26: Bathroom of Mrs. Lockwood's Room**

This is a carbon coated sample. Analysis was done on the second beige layer. It contains lead sulfate and zinc oxide. No baryite was identified.





**Figure 114: X-Ray Energy Dispersive Analysis of Sample 26: Bathroom of Mrs. Lockwood's Room**

This analysed area is the same area as in figure 113. They have almost the same results.

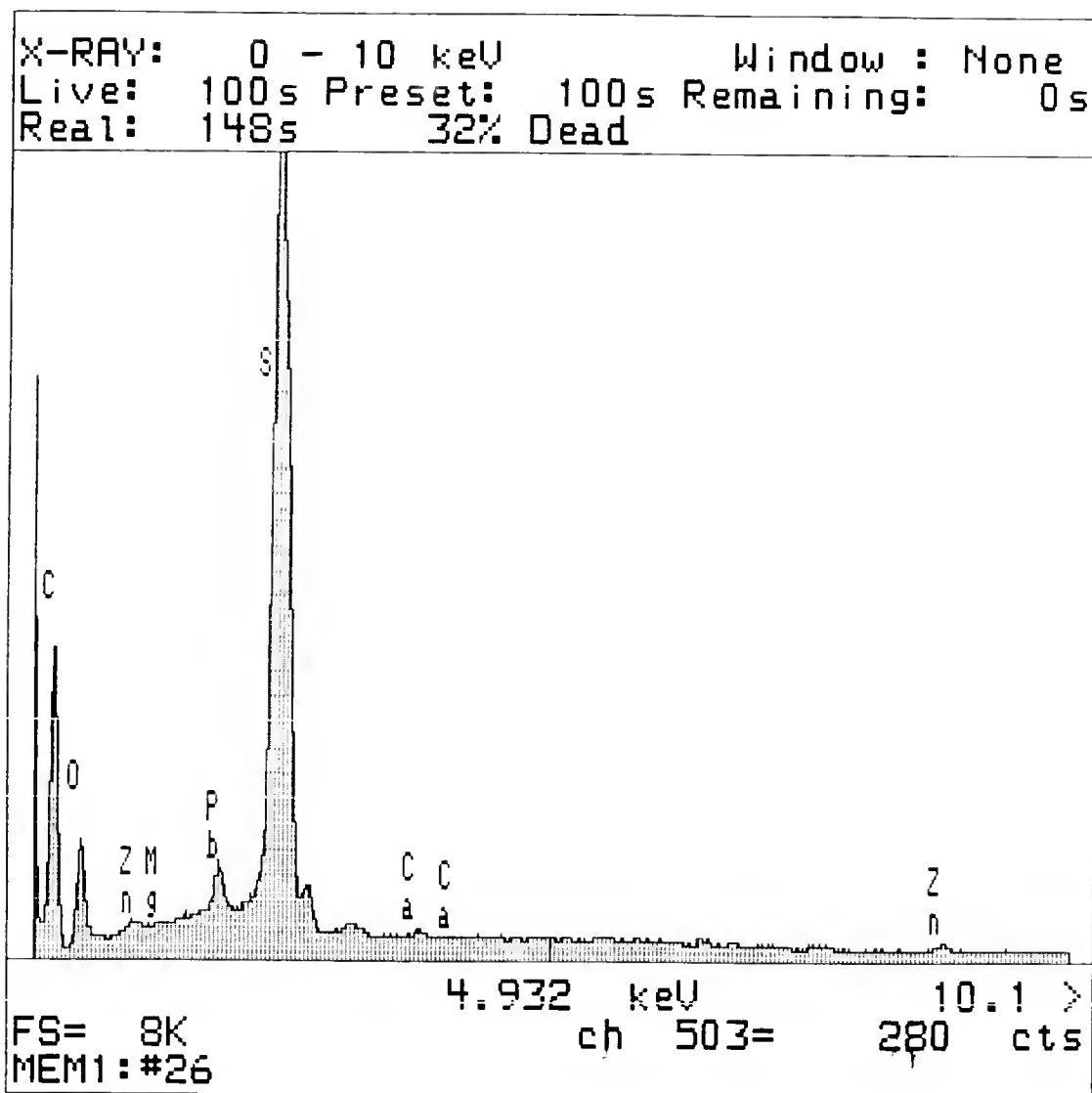


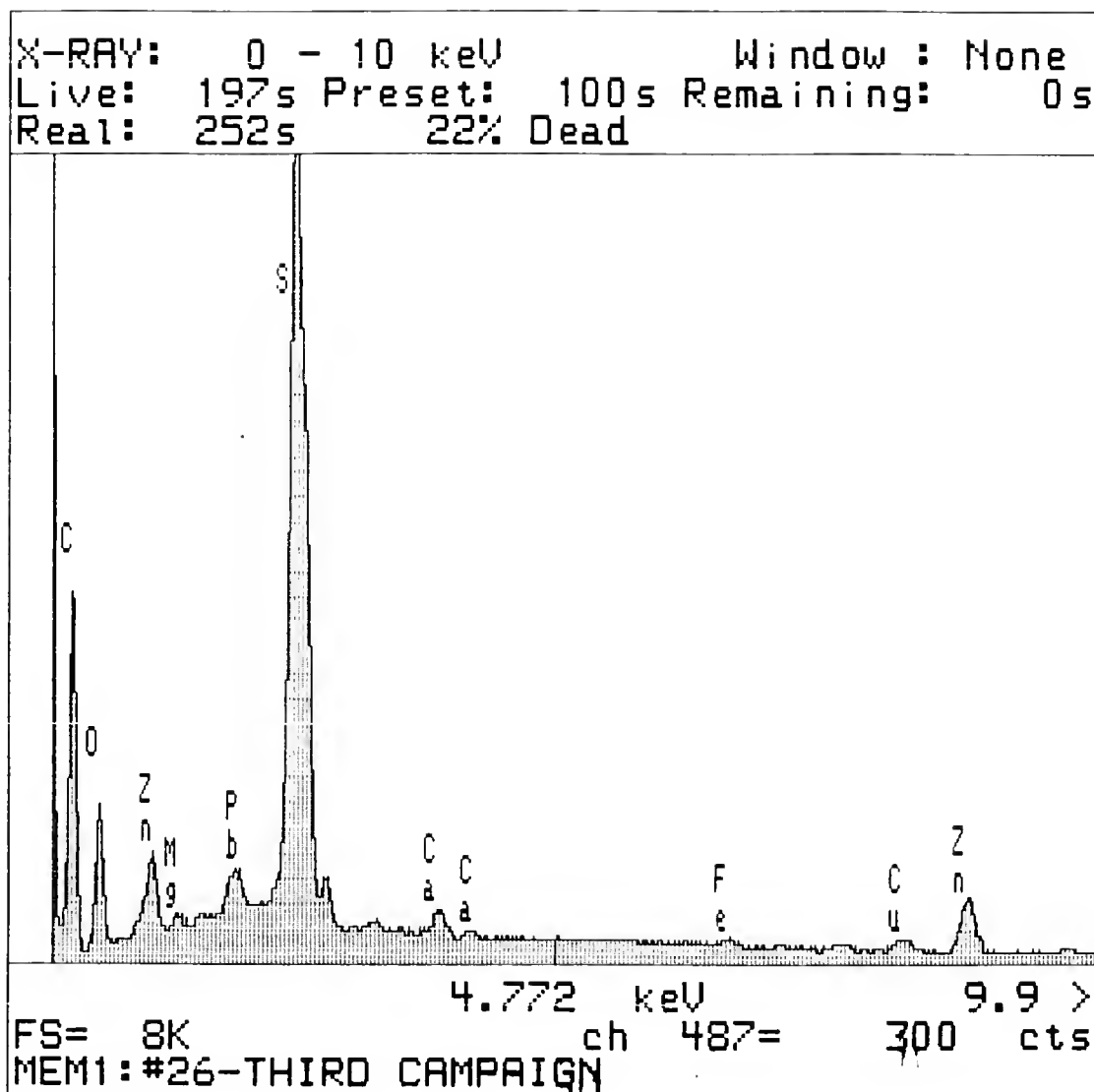




Figure 115: X-Ray Energy Dispersive Analysis of Sample 26: Bathroom of Mrs.

Lockwood's Room

The third campaign has some iron oxide, lead sulfate, and zinc oxide.





**Figure 116: Photo Micrograph of Sample 27: Bathroom of Mrs. Lockwood's Room**

**Sample Location:** Side of bathtub

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 27

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**





**Figure 117: X-Ray Energy Dispersive Analysis of Sample 27: Bathroom of Mrs. Lockwood's Room**

Carbon coated sample. The beige white layer has no lead but a high amount of zinc. Must be zinc white. Chlorine indicates the presence of an organic yellow pigment.

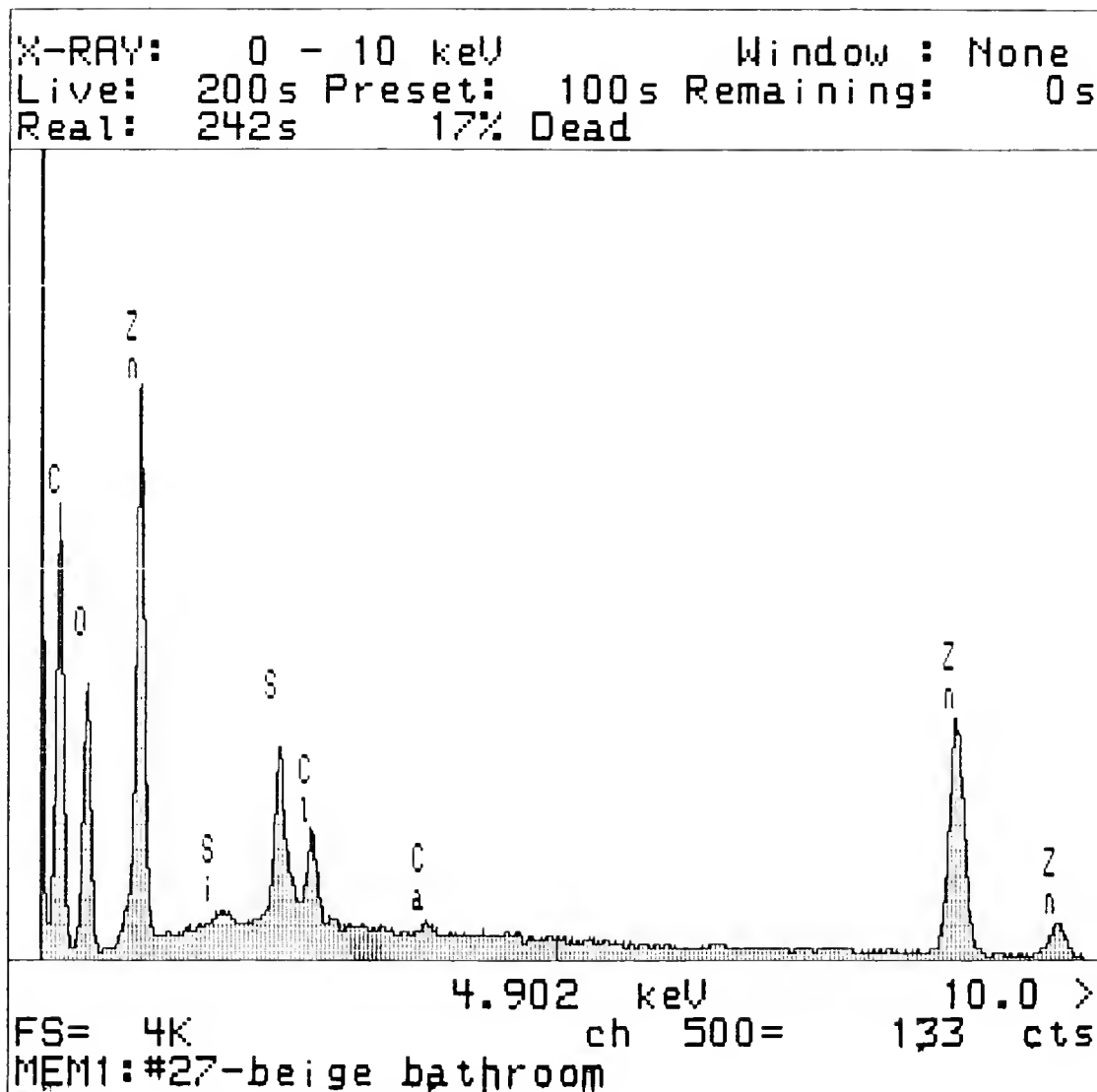




Figure 118: X-Ray Energy Dispersive Analysis of Sample 27: Bathroom of Mr.

Lockwood's Room

The carbon coated sample indicated that the gold leaf was used. Zn and Cr were also found.

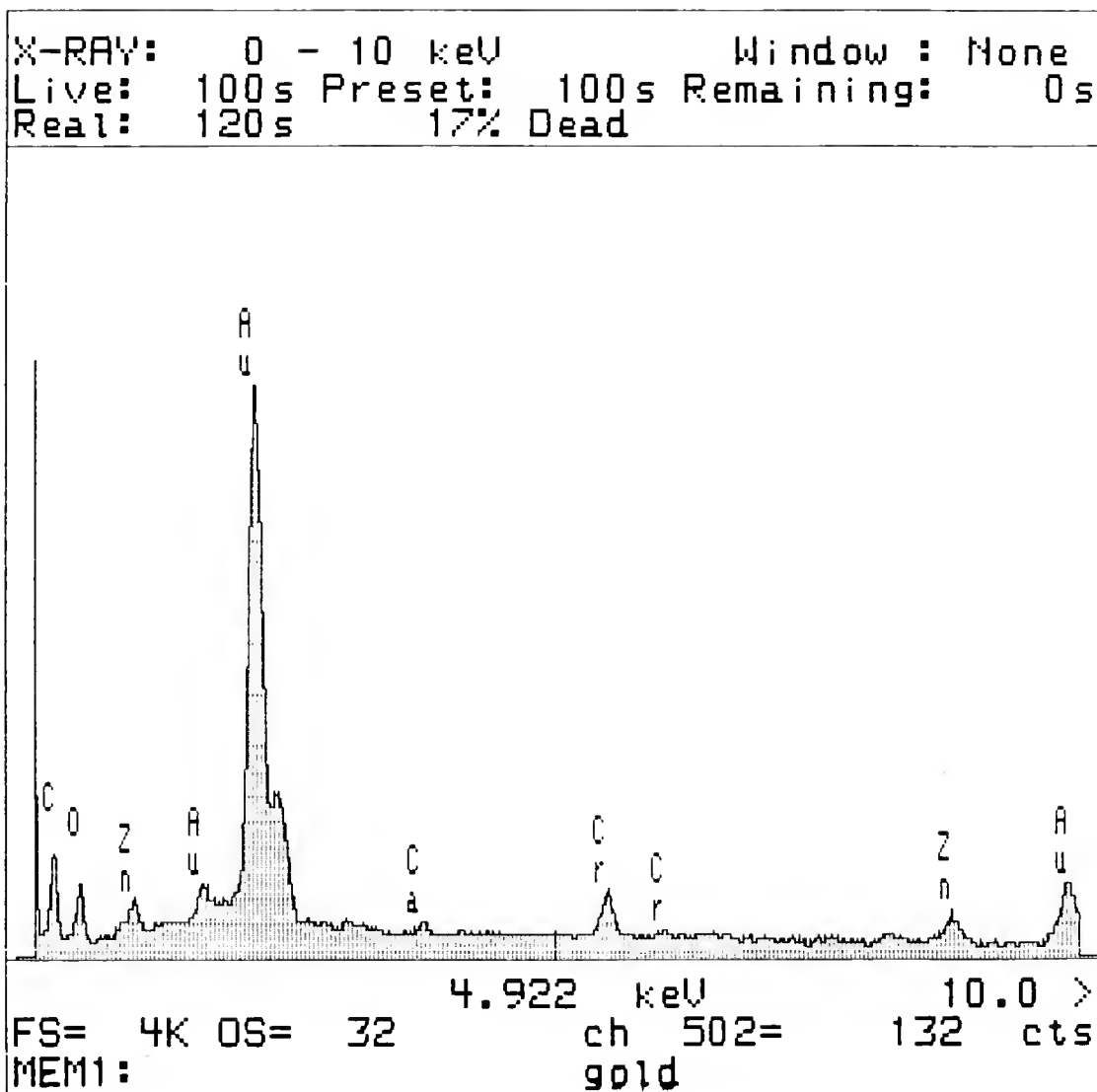
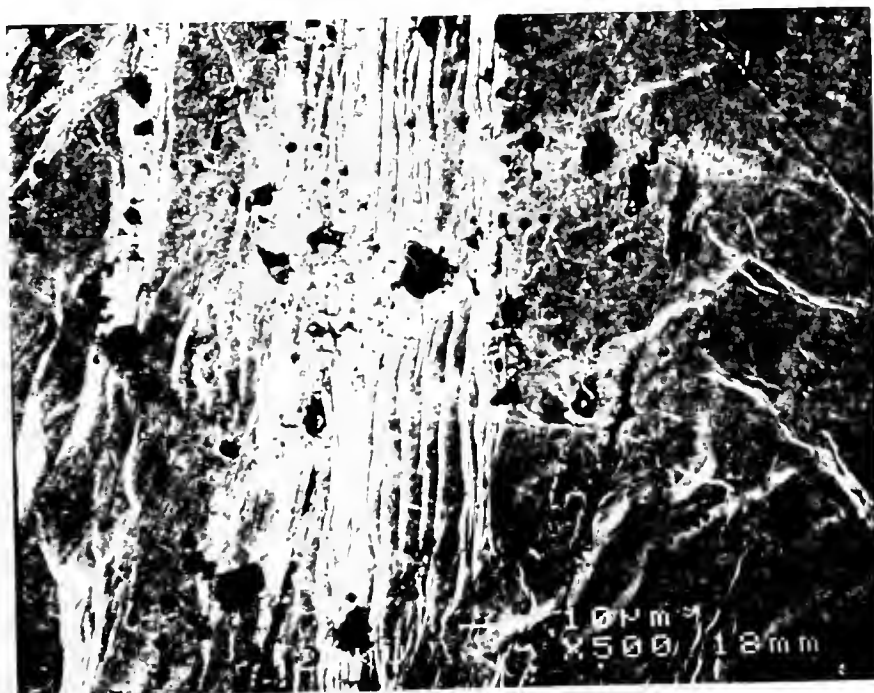






Figure 119: Backscatter Electron Image of the Gold layer in the Bathroom of Mrs. Lockwood's Room





## APPENDIX D: MR. LOCKWOOD'S ROOMS

Figure 120: Photograph of Sample 30 Area: Connecting Room to Mrs. Lockwood's Room





**Figure 121: Photo Micrograph of Sample 30A: Connecting Room to Mrs.**

**Lockwood's Room**

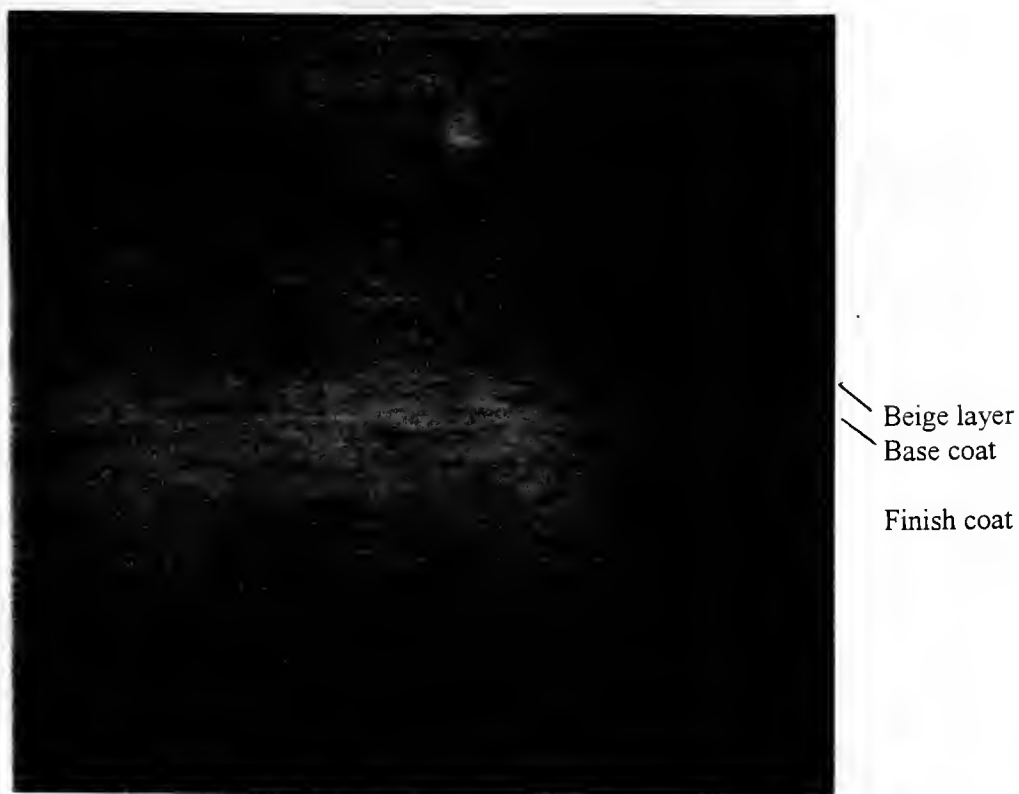
**Sample Location: Ceiling**

**Type of Film: 200 ASA Kodak Royal Gold, Film 2 Negative 34**

**Camera: Nikon**

**Magnification: 125X**

**Reflected light**





**Figure 122: Photo Micrograph of Sample 30B: Connecting Room to Mrs.  
Lockwood's Room**

**Sample Location:** Cornice

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 35

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**







**Figure 123: Photo Micrograph of Sample 30C: Connecting Room to Mrs. Lockwood's Room**

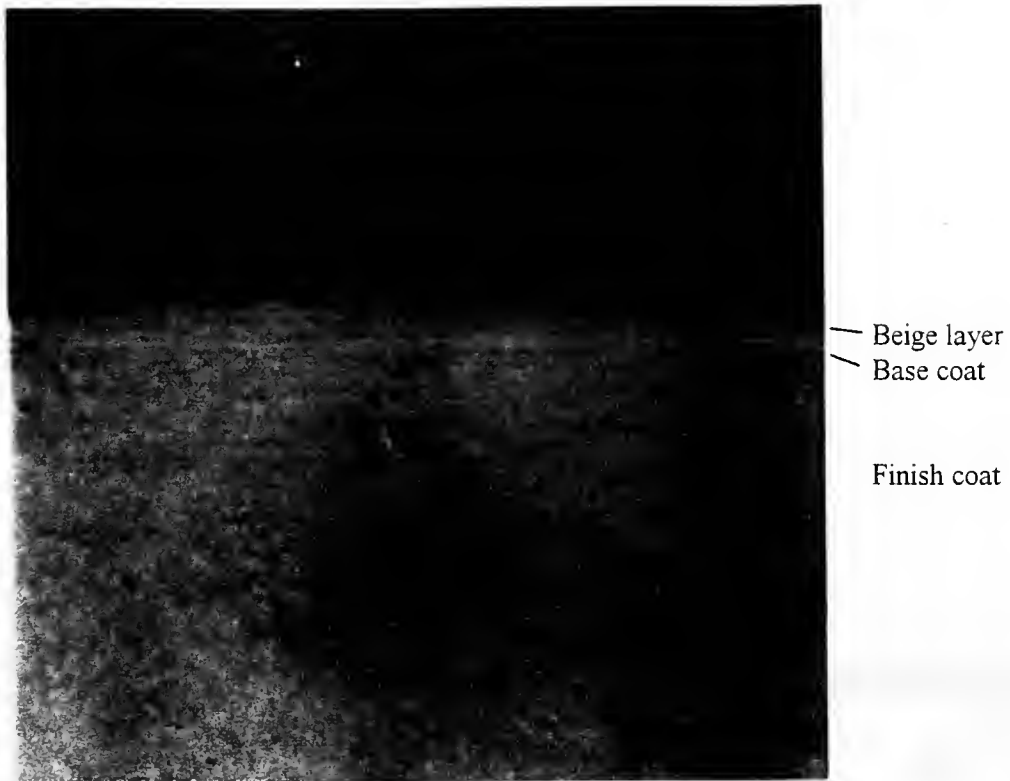
**Sample Location:** Wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 27

**Camera:** Nikon

**Magnification:** 125X

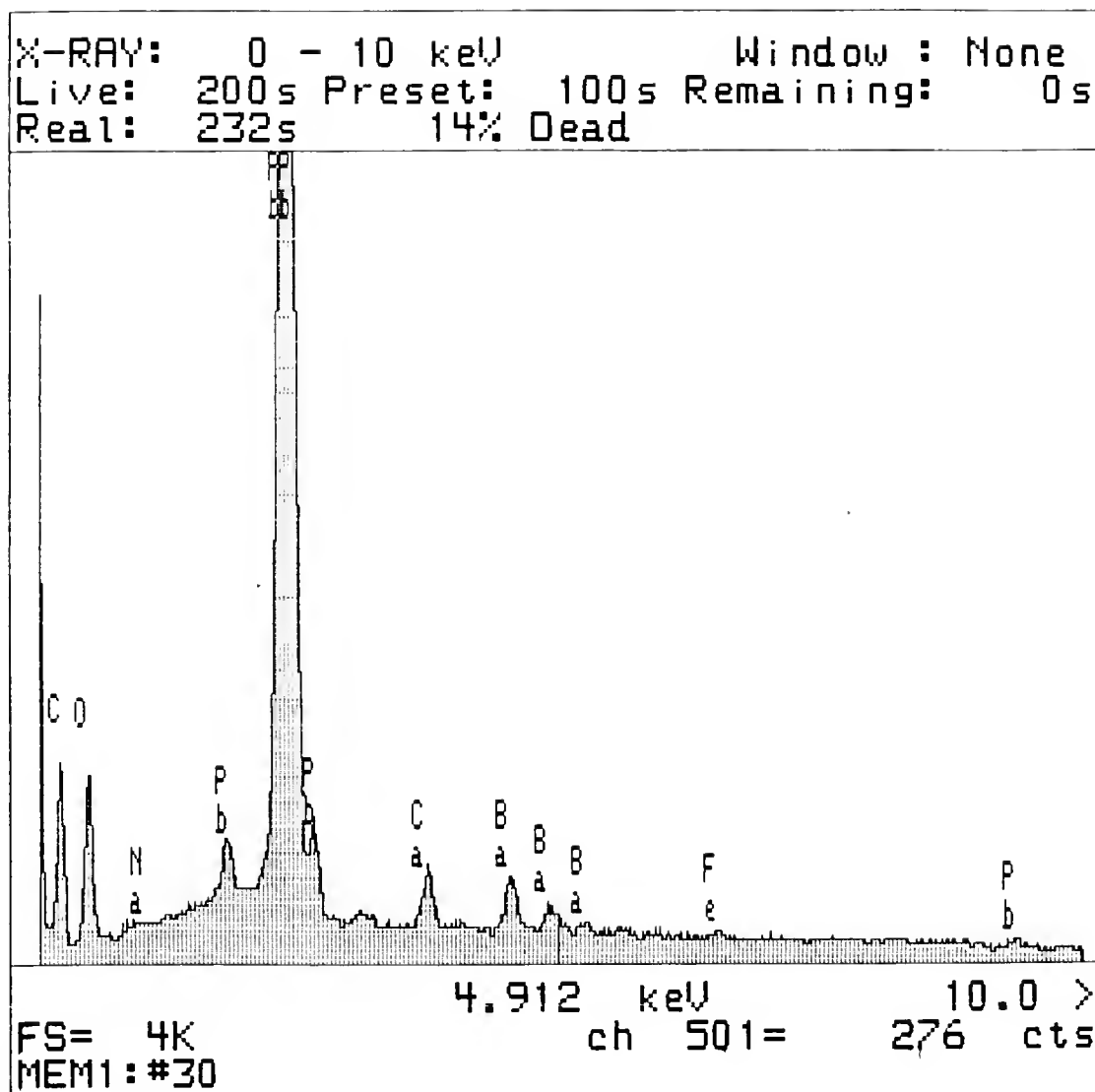
**Reflected light**





**Figure 124: X-Ray Energy Dispersive Analysis of Sample 30A: Connecting Room to Mrs. Lockwood's Room**

Sample is from the beige area. Possible yellow lead oxide, carbon black, yellow ochre, and calcium carbonate.





**Figure 125: Photograph of Sample 32 Area: Mr. Lockwood's Room**



**Figure 126: Photo Micrograph of Sample 32: Mr. Lockwood's Room**

**Sample Location:** Wall, northeast corner near ventilation

**Type of Film:** 200 ASA Kodak Royal Gold, Film 2 Negative 37

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

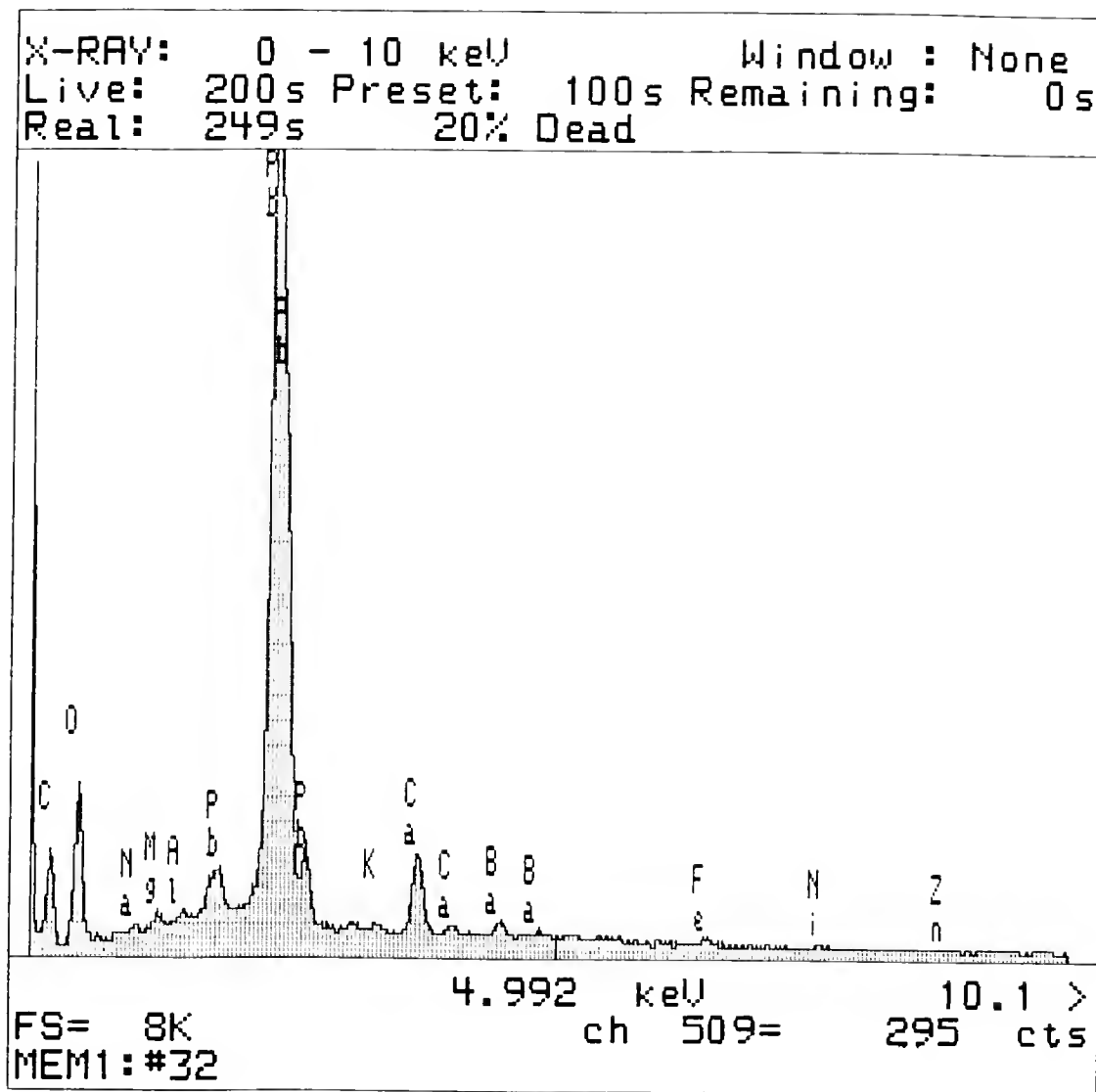


— Greenish layer  
— Base coat



Figure 127: X-Ray Energy Dispersive Analysis of Sample 32: Mr. Lockwood's Rm

This carbon coated sample of the green painted area consist of green earth, lithopone, and some organic pigment. Ni might indicate an inorganic pigment.







**Figure 128: Photo Micrograph of Sample 33: Mr. Lockwood's Room**

**Sample Location:** East wall, repainted area

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 3

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



Modern light pink  
paint layer.

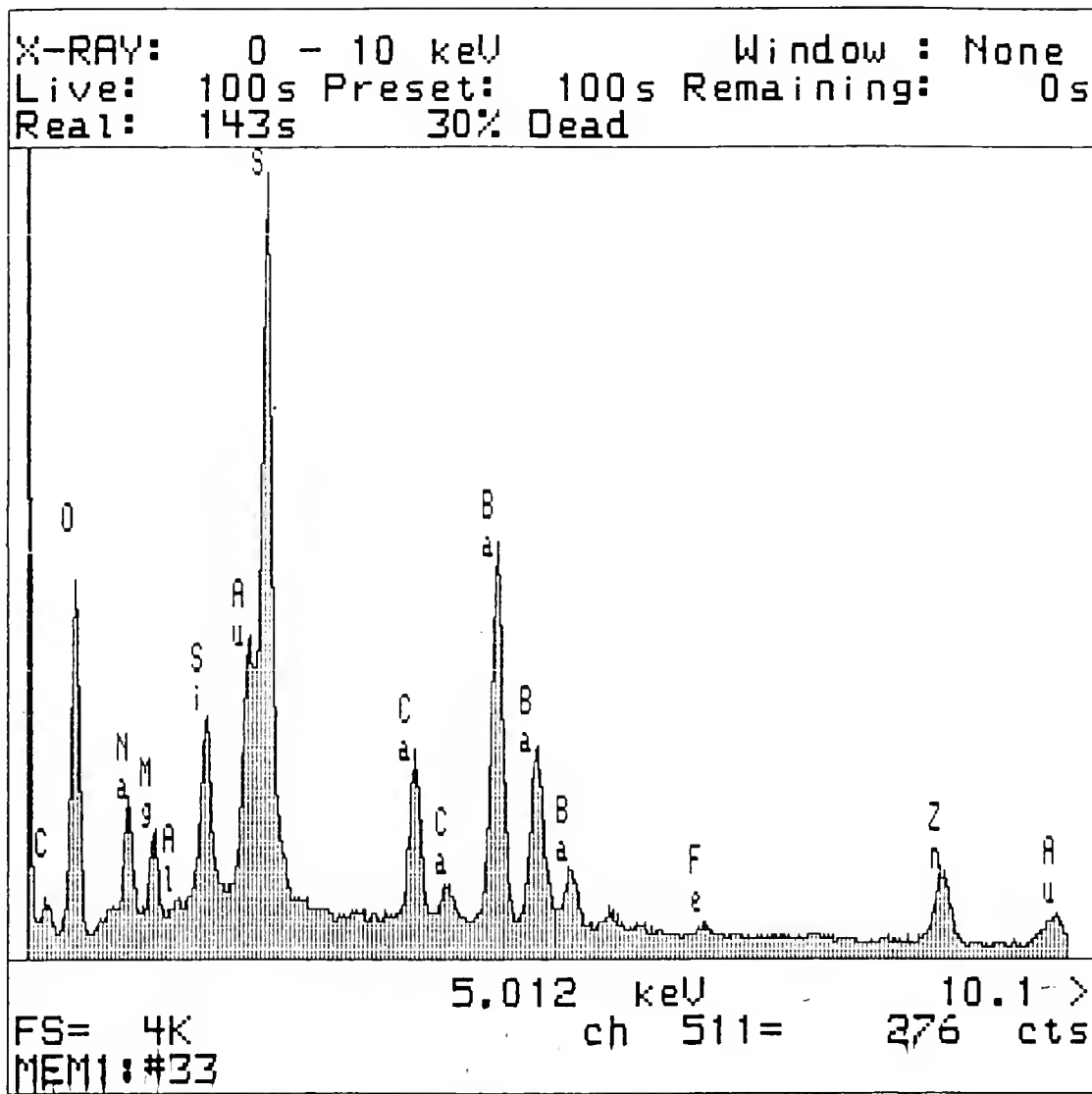
Original paint layer

Finish coat



Figure 129: X-Ray Energy Dispersive Analysis of Sample 33: Mr. Lockwood's Rm

This gold coated sample of the light pink modern paint consist of no lead. It has baryite, some iron oxides and zinc oxide. There is possibly alizarin crimson and lithapone.





**Figure 130: Photo Micrograph of Sample 34: Mr. Lockwood's Room**

**Sample Location:** East wall, repainted area

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 5

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



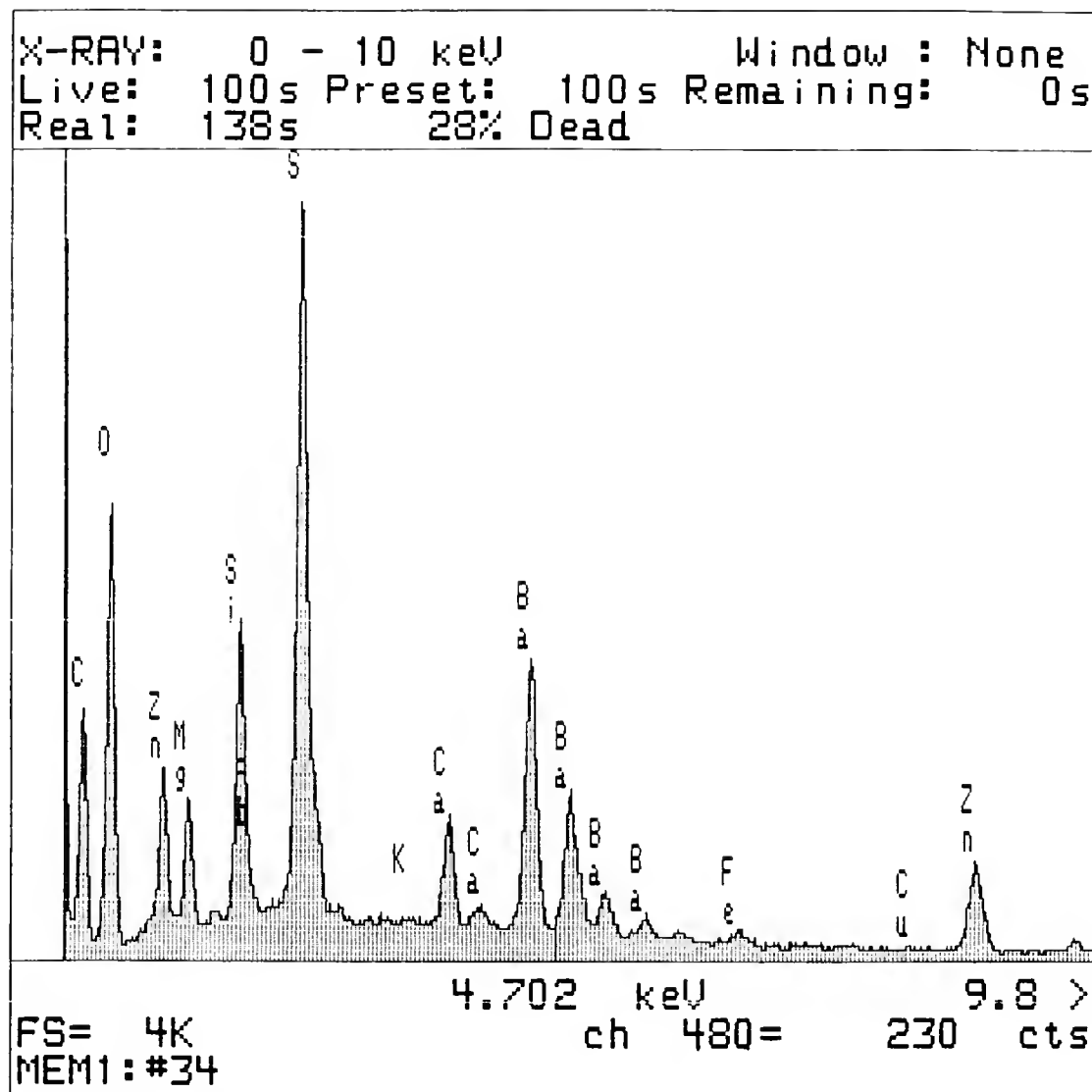
Modern green and  
grey paint layers. No  
original paint layer



Figure 131: X-Ray Energy Dispersive Analysis of Sample 34: Mr. Lockwood's Rm

This carbon coated sample of the repainted area indicates no titanium as was expected.

It has green earth, zinc oxide and baryte. Possible zinc green.







**Figure 132: Photograph of Sample 35 Area: Oratory of Mr. Lockwood's Room**



**Figure 133: Photo Micrograph of Sample 35A: Mr. Lockwood's Room**

**Sample Location:** East wall, repainted area

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 6

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



—Dark beige paint layer. Similar to Moorish Room

Base coat

—Finish coat



**Figure 134: Photograph of Sample 39 Area: Storage Room of Mr. Lockwood's Rm**





**Figure 135: Photo Micrograph of Sample 39: Storage Room of Mr. Lockwood's Rm**

**Sample Location:** West wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 9

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

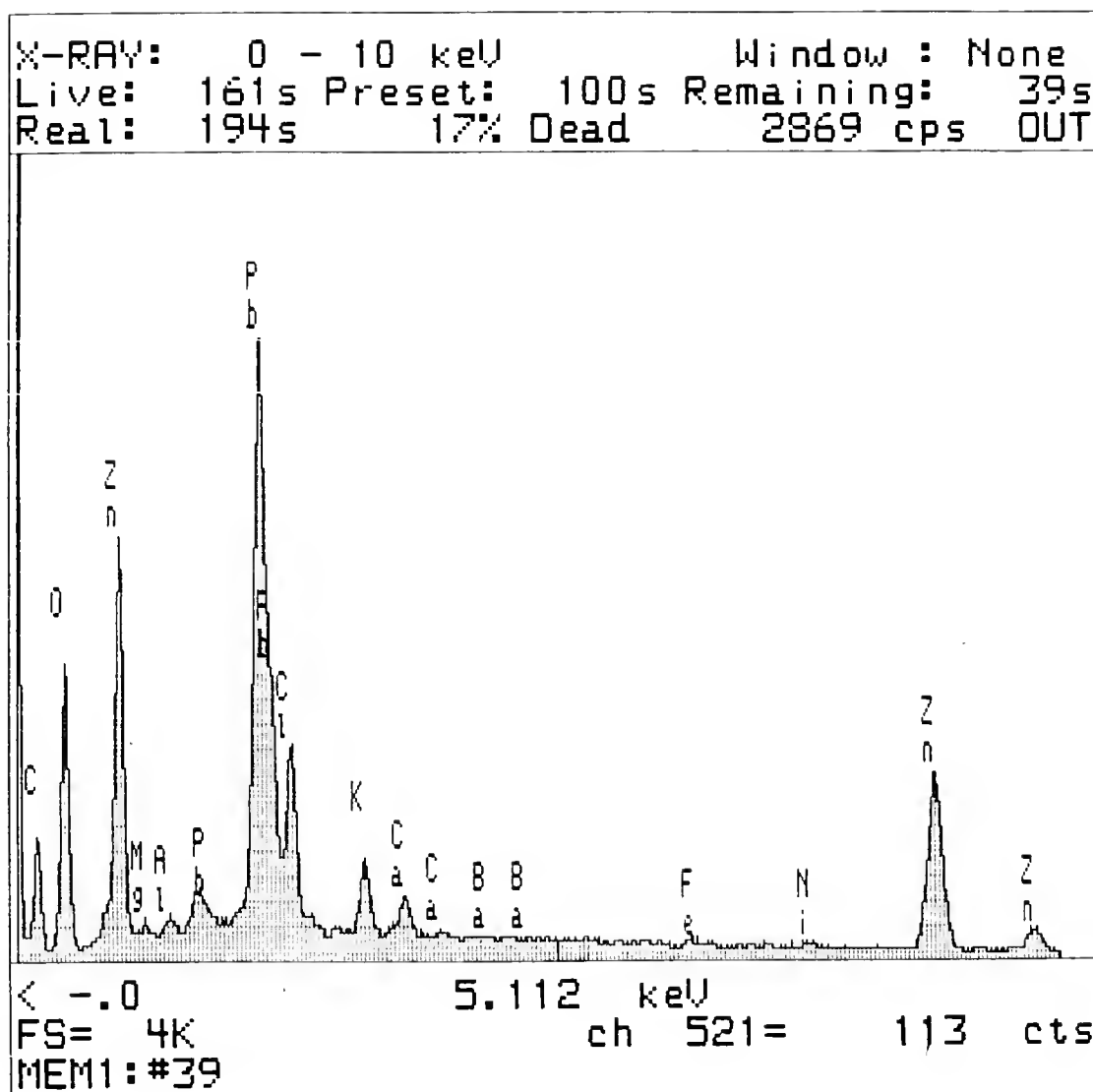


- Green paint layer.
- Base coat or original paint layer?
- Finish coat



**Figure 136: X-Ray Energy Dispersive Analysis of Sample 39: Storage Room of Mr. Lockwood's Room**

The carbon coated sample of the green paint layer revealed that it contains lead oxide, zinc oxide, and green earth. The presence of Cl and Ni indicates use of an organic blue pigment. It seems that this paint layer is not the original paint.







**Figure 137: Photograph of Sample 36 Area: Mr. Lockwood's Bathroom**





**Figure 138: Photo Micrograph of Sample 36: Mr. Lockwood's Bathroom**

**Sample Location:** Inside closet

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 2

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

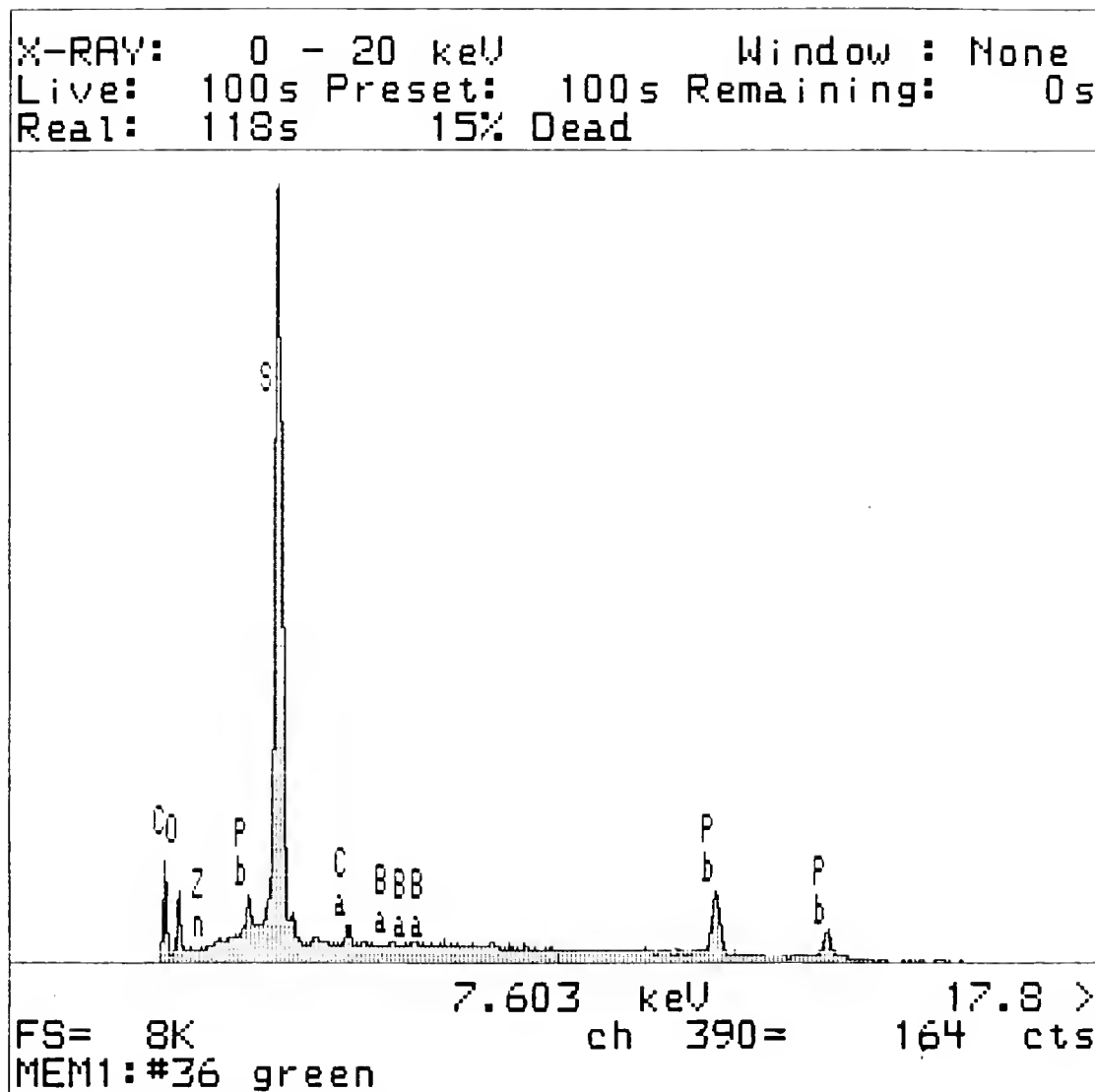


- Greenish layer,  
  similar to sample 39
- Base coat
- Finish coat



**Figure 139: X-Ray Energy Dispersive Analysis of Sample 36: Mr. Lockwood's Bathroom**

This carbon coated sample of the green painted area contains baryite, zinc oxide, maybe yellow lead, and/or zinc green.





## APPENDIX E: MOORISH ROOM

**Figure 140: Sample 37 and 38 Area: Moorish Room**



**Figure 141: Photo Micrograph of Sample 37: Moorish Room**

**Location:** Wall above east door

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 4

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



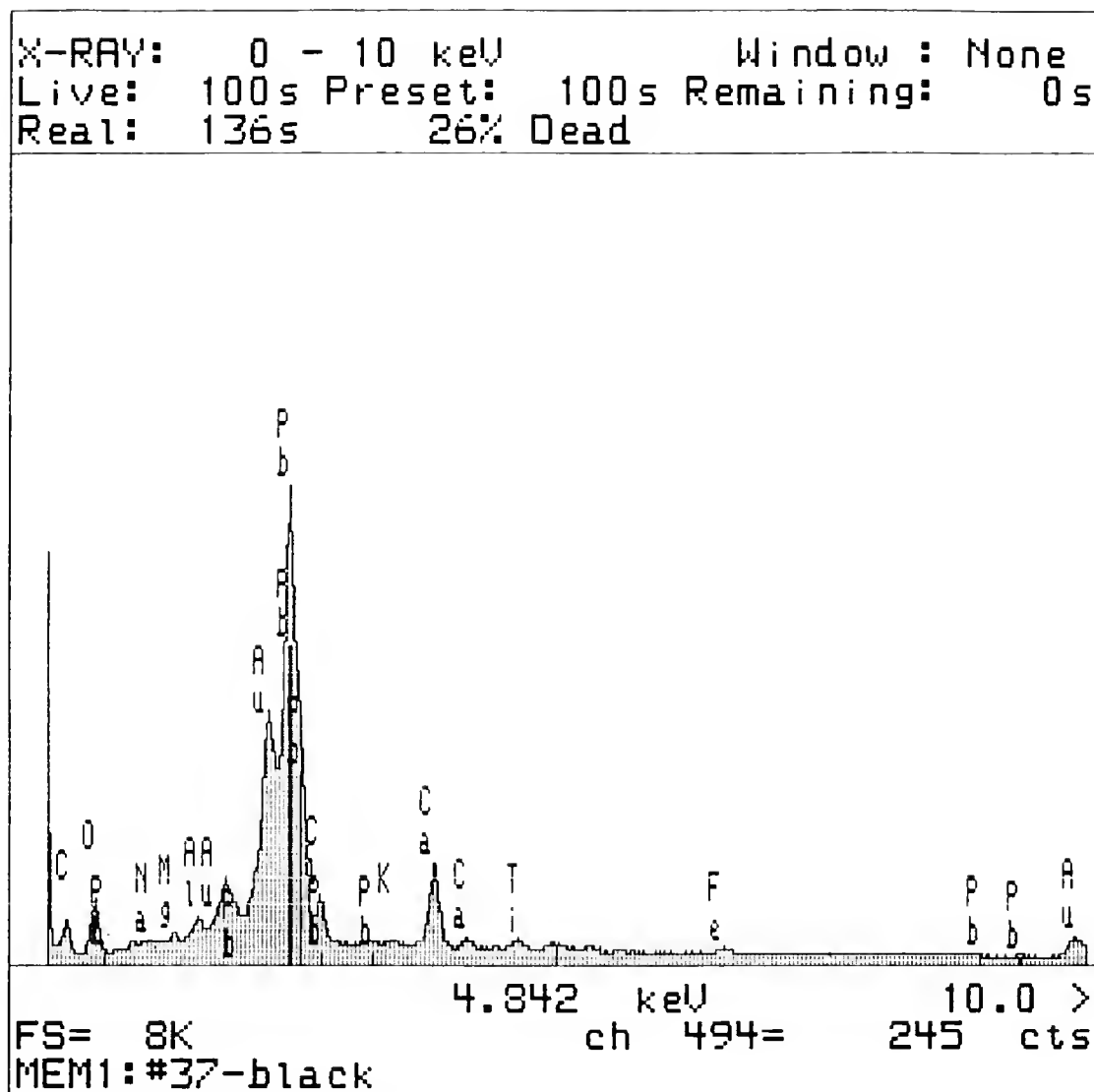
— Gold and Black  
— campaign  
— Field paint layer  
— Base coat  
— Finish coat





**Figure 142: X-Ray Energy Dispersive Analysis of Sample 37: Moorish Room**

This gold coated sample of the black painted area indicates the presence of carbon black, and lead blacks. It possible that some organic pigments are present.





**Figure 143: Photo Micrograph of Sample 38A and B: Moorish Room**

**Location:** Wall above east door

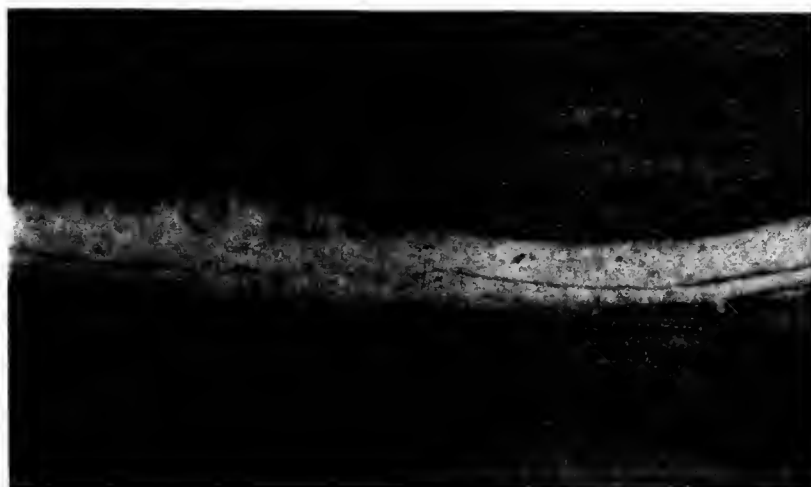
**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 6, Film 3 Negative 7

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

Sample 38A: Black and grey



Gold and grey  
campaign  
Field paint layer  
Base coat

Sample 38 B: Orange and beige

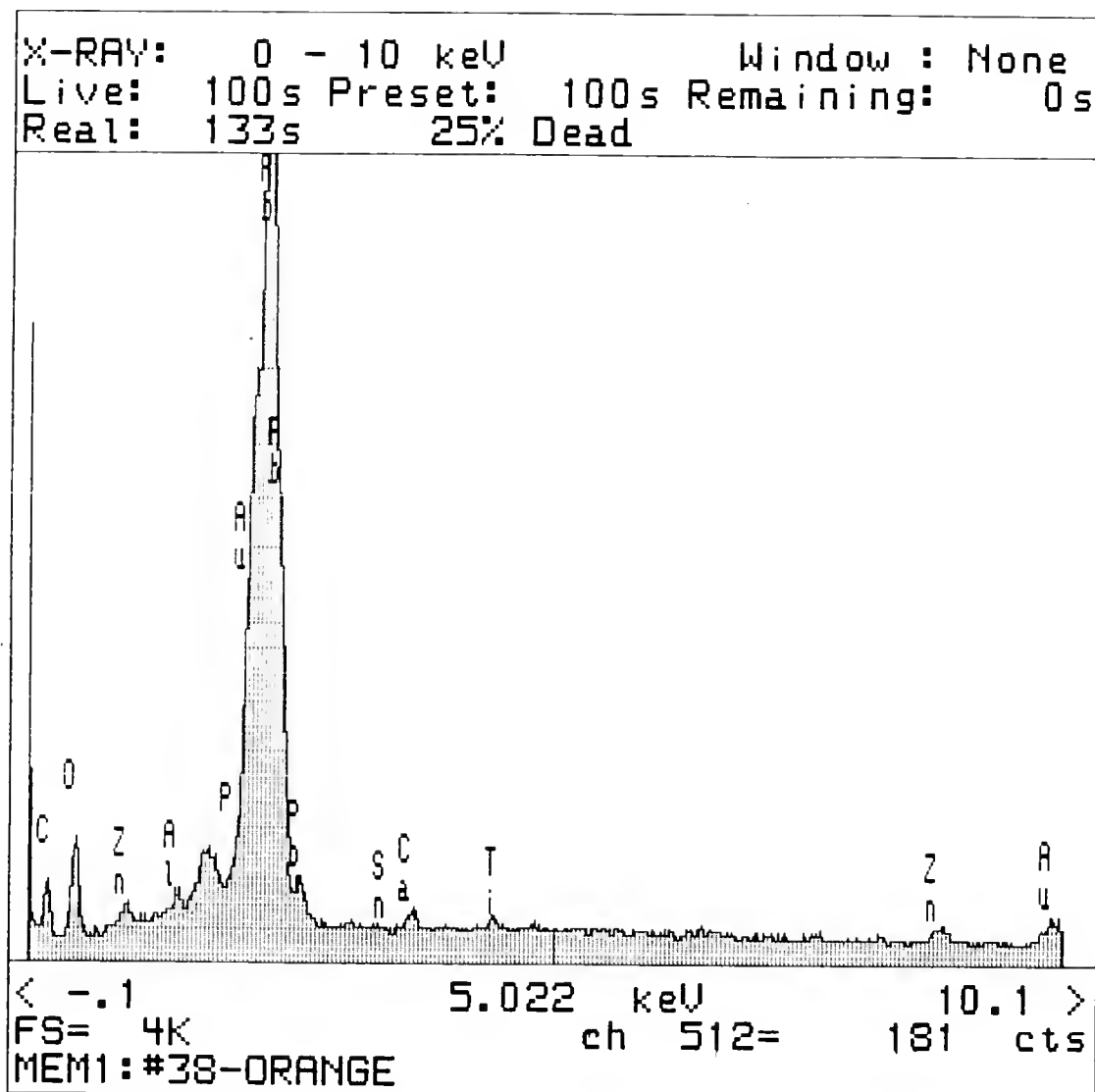


Orange and beige  
campaign  
Field paint layer  
Base coat



**Figure 144: X-Ray Energy Dispersive Analysis of Sample 38: Moorish Room**

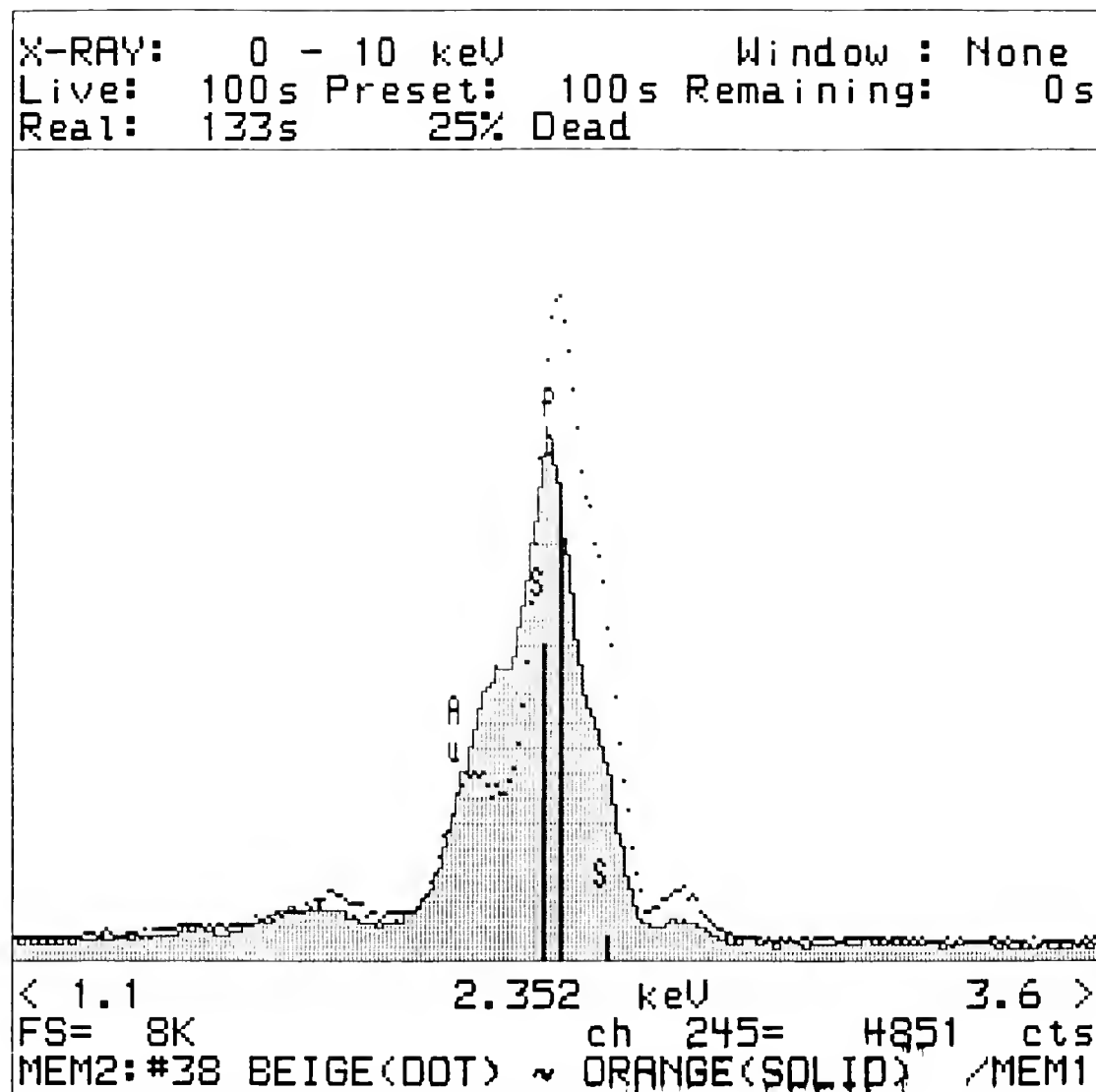
In this gold coated sample, lead and sulphur is present in the orange painted area. It is not conclusive that mercury is present, but more X-Ray energy dispersive analysis revealed that vermilion was used. It seems that red lead could have been used.





**Figure 145: X-Ray Energy Dispersive Analysis of Sample 38: Moorish Room**

This is the same sample as in figure 144. The only difference found was the that the beige has more sulphur in it than the orange.

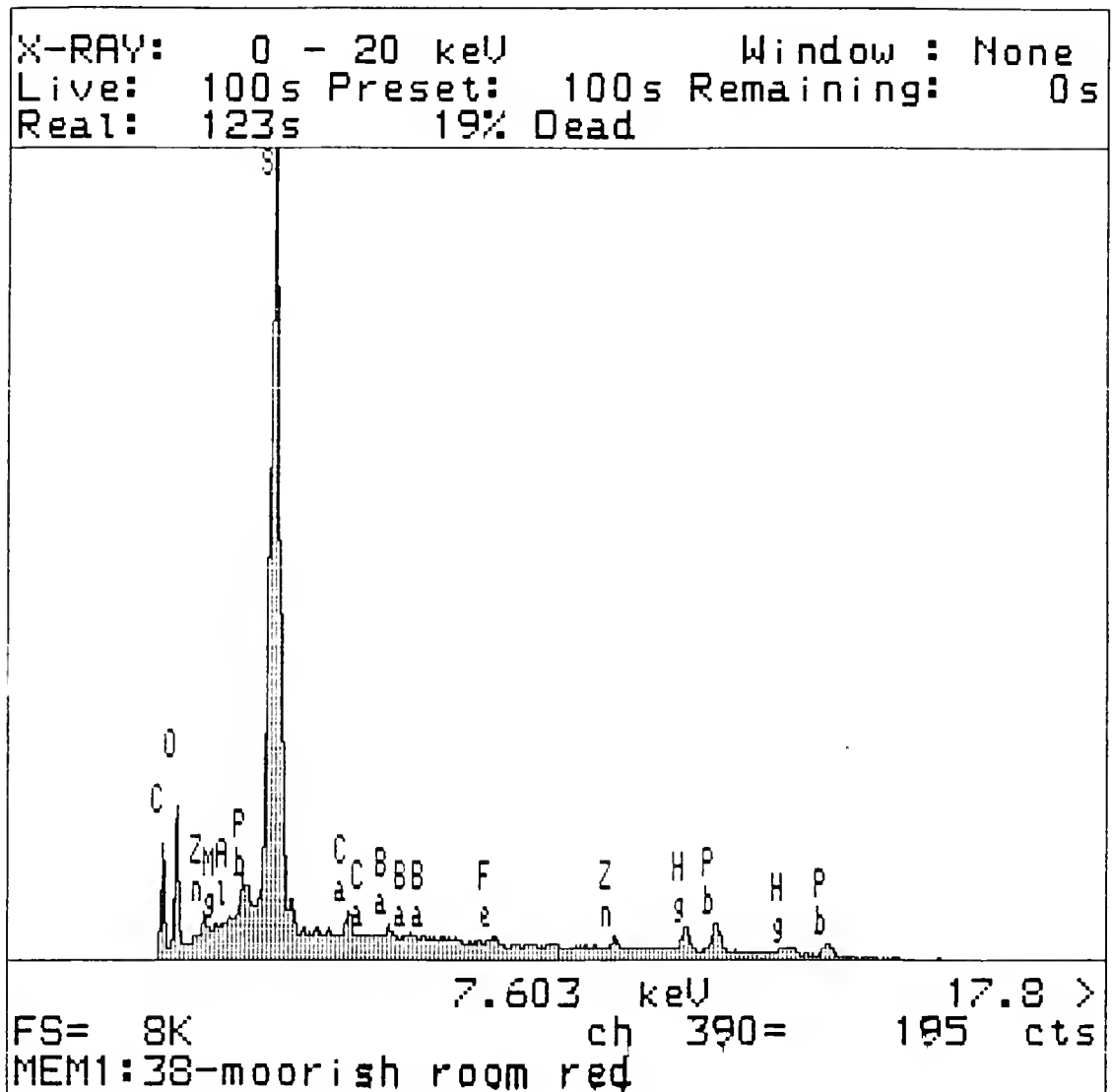






**Figure 146: X-Ray Energy Dispersive Analysis of Sample 38: Moorish Room**

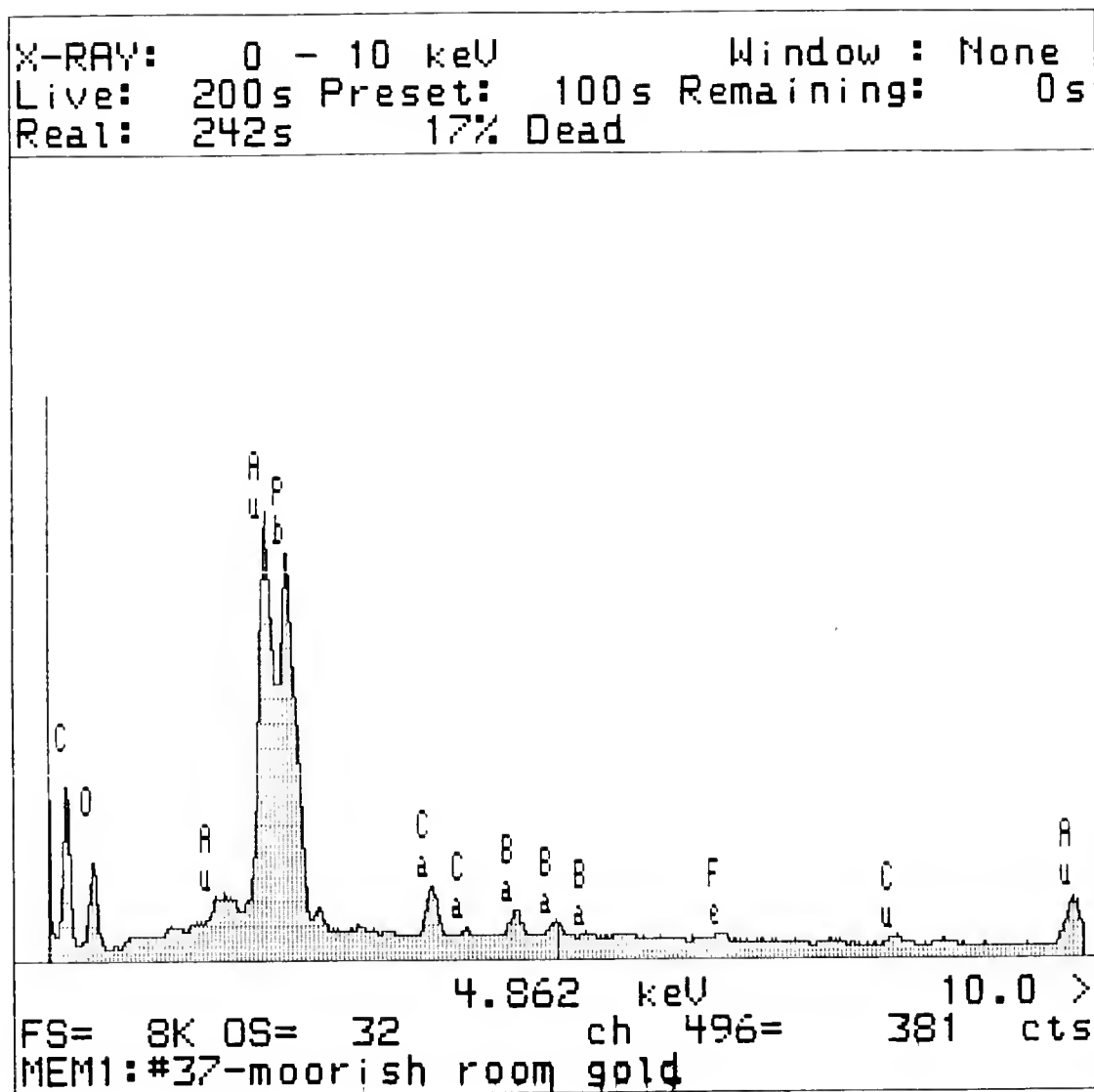
This is a carbon coated red paint layer. Vermillion is used as a pigment. Lithopone is present.





**Figure 147: X-Ray Energy Dispersive Analysis of Sample 37: Moorish Room**

This carbon coated sample of the gold layer consists of gold, a small amount of copper and iron. It is gold Leaf.

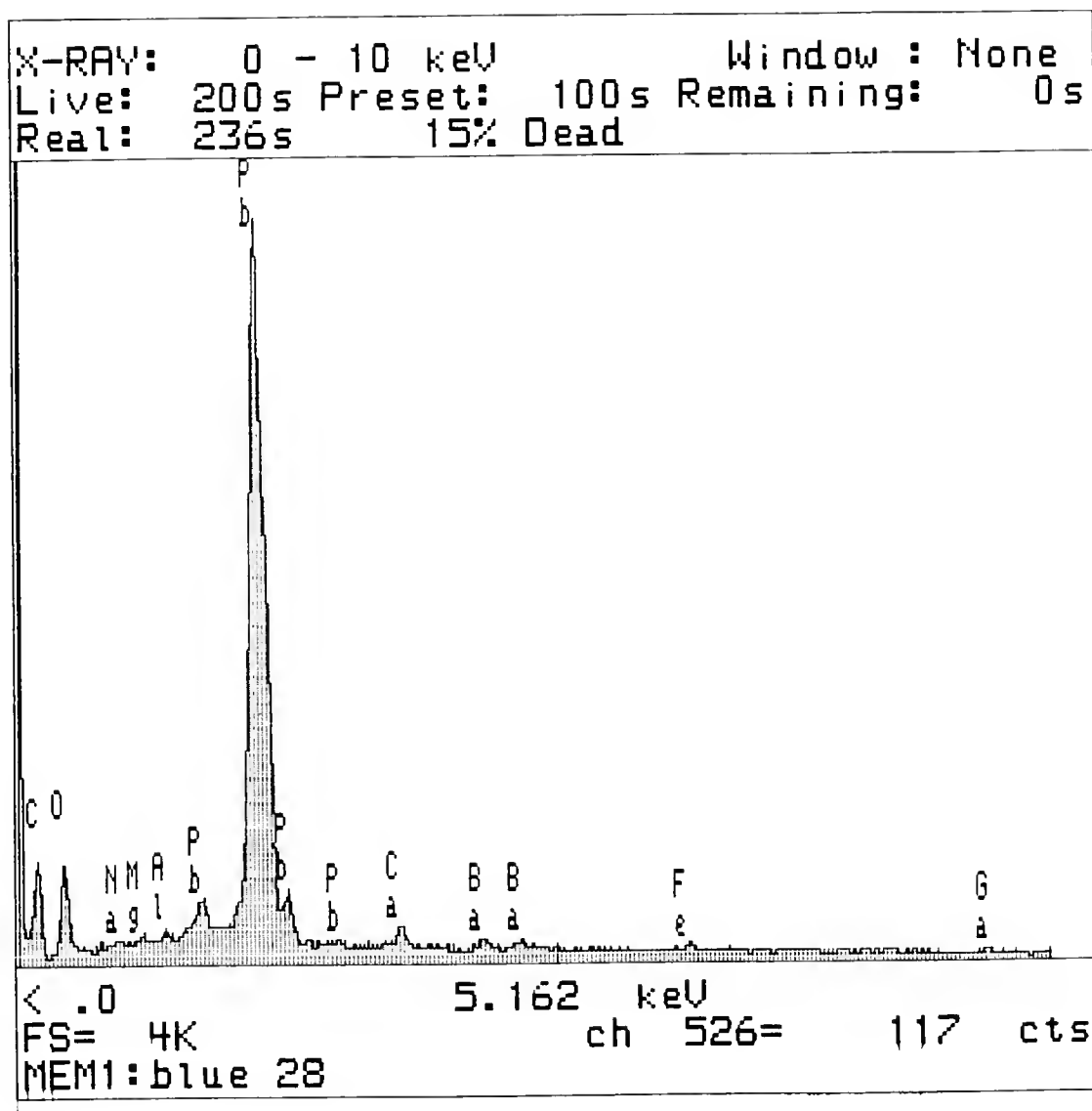




## APPENDIX F: Hallway

**Figure 148: X-Ray Energy Dispersive Analysis of Sample 28: Hallway**

The carbon coated sample of the blue painted area reveals a mixture of possibly ultramarine and prussian blue. (Fig. 24)





## APPENDIX G: SECONDARY STAIRCASE

Figure 149: Photograph of Sample 41 Area: Secondary Staircase







**Figure 150: Photo Micrograph of Sample 41: Secondary Staircase**

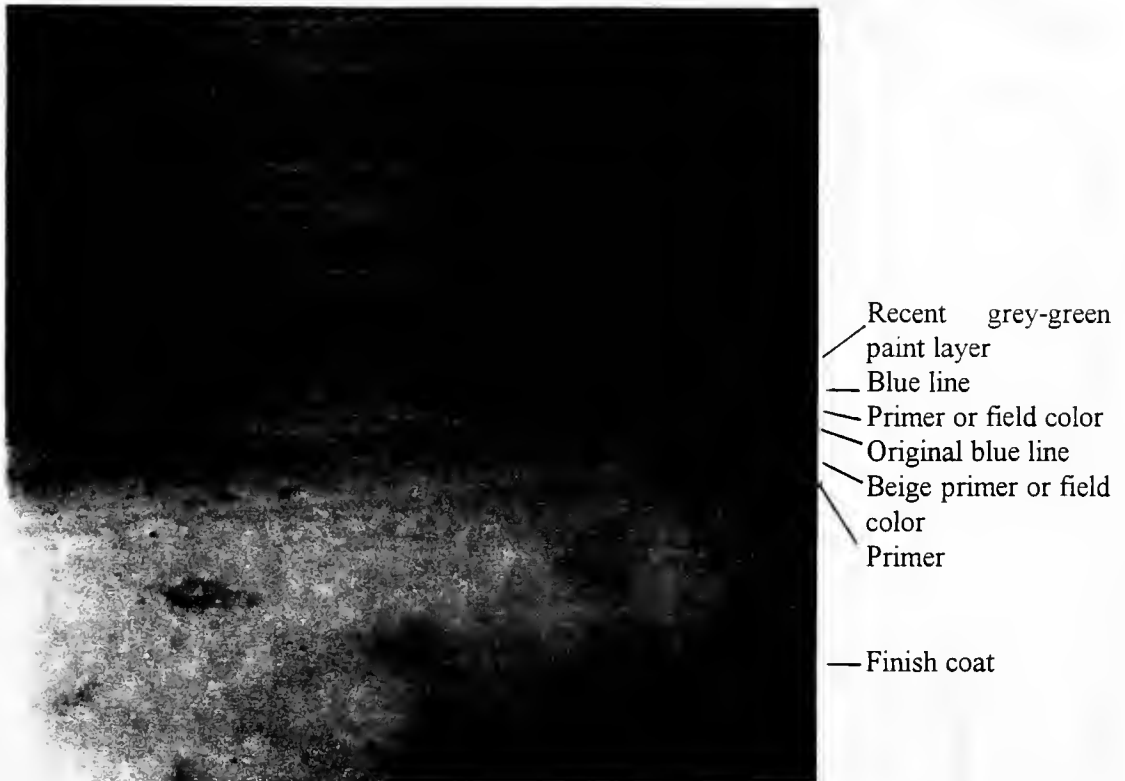
**Location:** Third floor on blue stripe on wall

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 11A

**Camera:** Nikon

**Magnification:** 125X

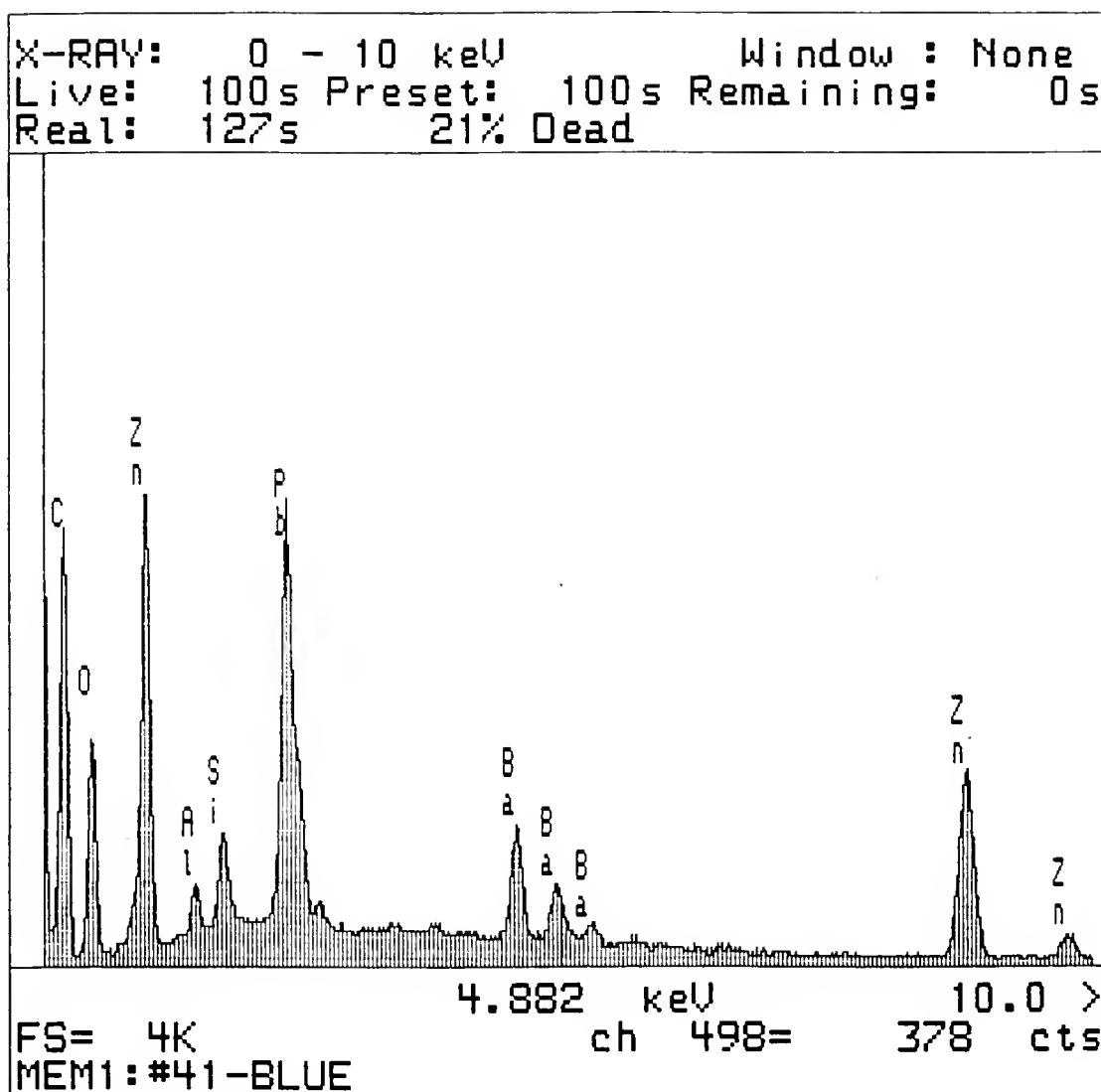
**Reflected light**





**Figure 151: X-Ray Energy Dispersive Analysis of Sample 41: Secondary Staircase**

This is a carbon coated sample of the second blue line. It contains a high amount of zinc. Possible zinc oxide or lead oxide. The Al and Si indicates that ultramarine blue could have been used as the blue pigment.





## APPENDIX H: ITALIAN SUITE

**Figure 152: Photograph of Sample 40 Area: Italian Suite**



**Figure 153: Photo Micrograph of Sample 40: Italian Suite**

**Location:** Woodwork near window

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 10A

**Reflected Light**

**Camera:** Nikon

**Magnification:** 125X



Glazing, graining  
effect on prepared  
surface

Primer

Wood



## APPENDIX I: LIBRARY

**Figure 154: Photo Micrograph of Sample 55: Library**

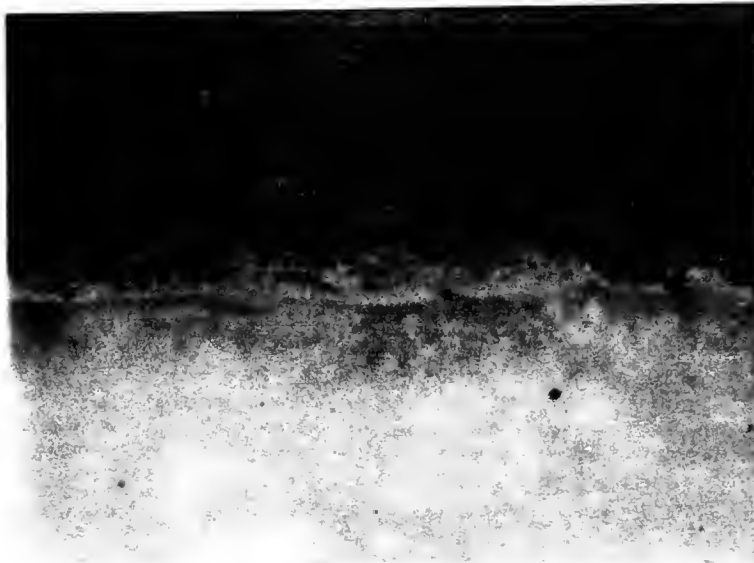
**Location:** Fragments from ceiling - Graining

**Type of Film:** 200 ASA Kodak Royal Gold, Film 5 Negative 6

**Reflected Light**

**Camera:** Nikon

**Magnification:** 125X



- Graining
- Base coat for graining, Primer
- Finish coat, note no quartz particles

**Figure 155: Photo Micrograph of Sample 56: Library**

**Location:** Fragments from ceiling - Graining

**Type of Film:** 200 ASA Kodak Royal Gold, Film 5 Negative 5

**Reflected Light**

**Camera:** Nikon

**Magnification:** 125X



- Gold layer
- Base coat for gold
- Finish coat





Figure 156: X-Ray Energy Dispersive Analysis of Sample 55: Library

This carbon coated sample of the brown painted area, shows no iron oxide as expected but carbon black with calcium carbonate and gypsum.

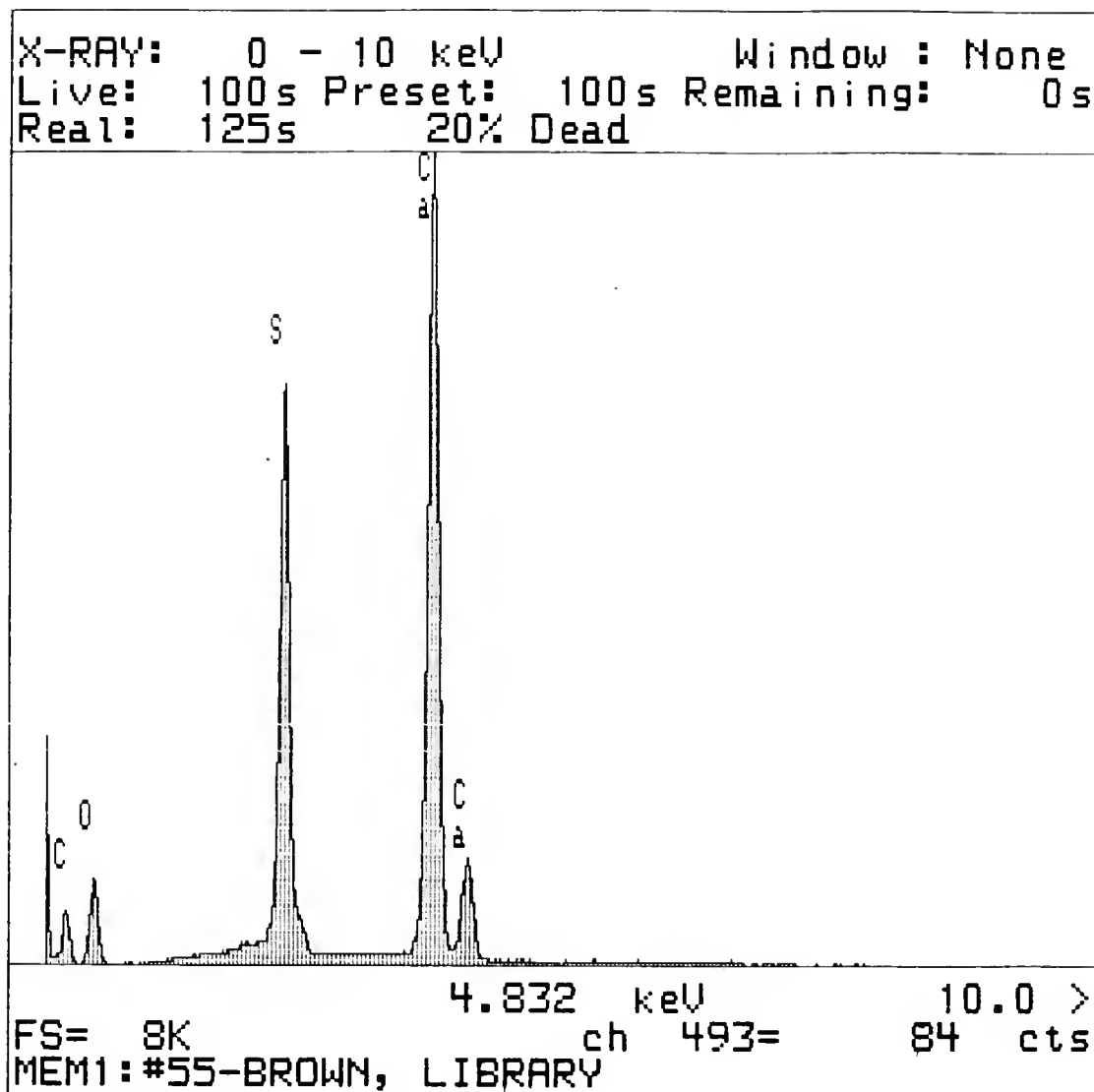
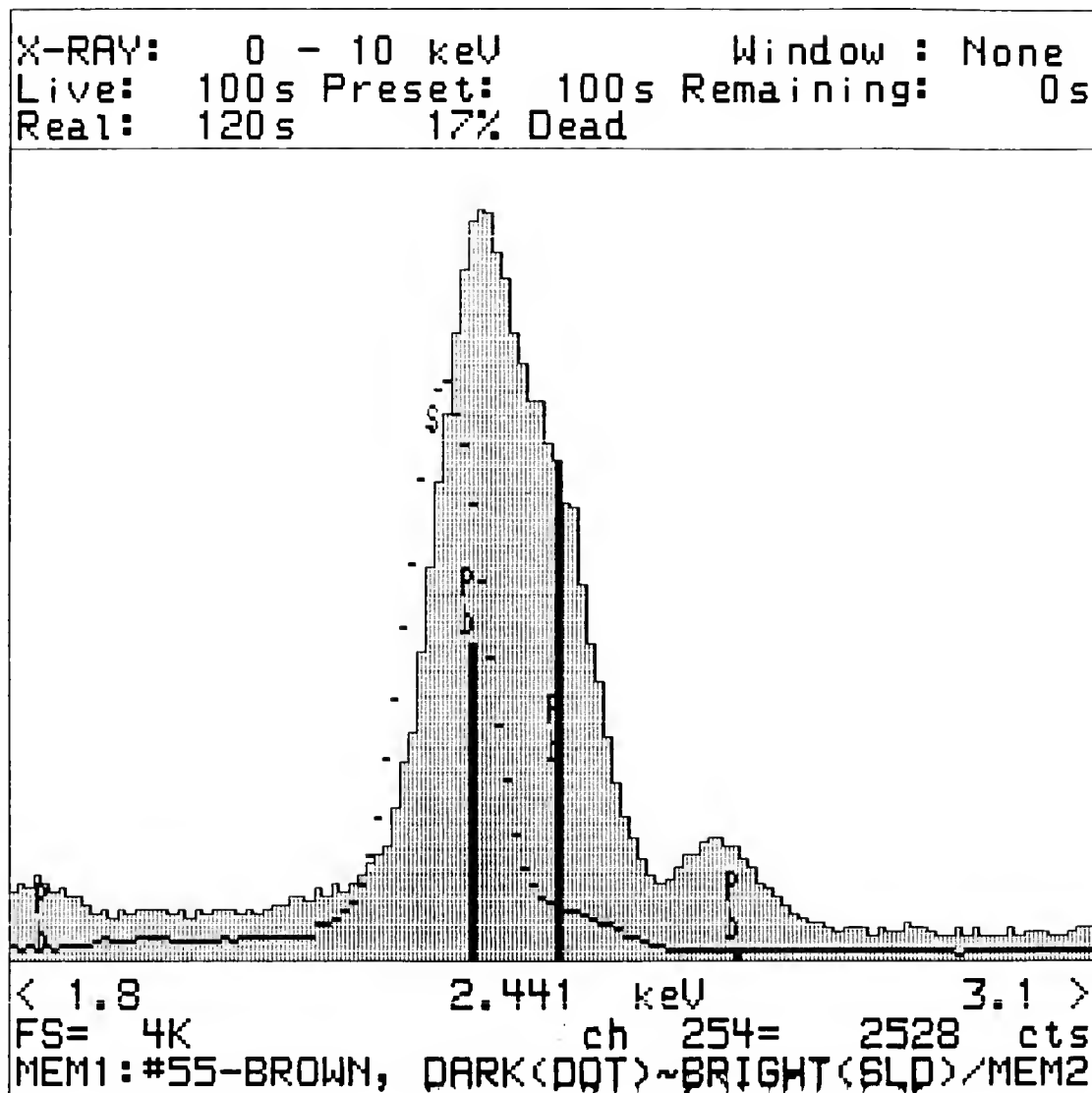




Figure 157: X-Ray Energy Dispersive Analysis of Sample 55: Library

This is the same sample as in Figure 156. It is a comparison between the dark and light areas on the BEI. Lead and sulphur are concentrated in different areas.





## APPENDIX J: ROTUNDA

**Figure 158: Photo Micrograph of Sample 51: Rotunda**

**Location:** Fragments from wall, Lockwood period

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 21A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**



Dark pink layer

Base coat

Sizing

Finish coat



**Figure 159: Photo Micrograph of Sample 52: Rotunda**

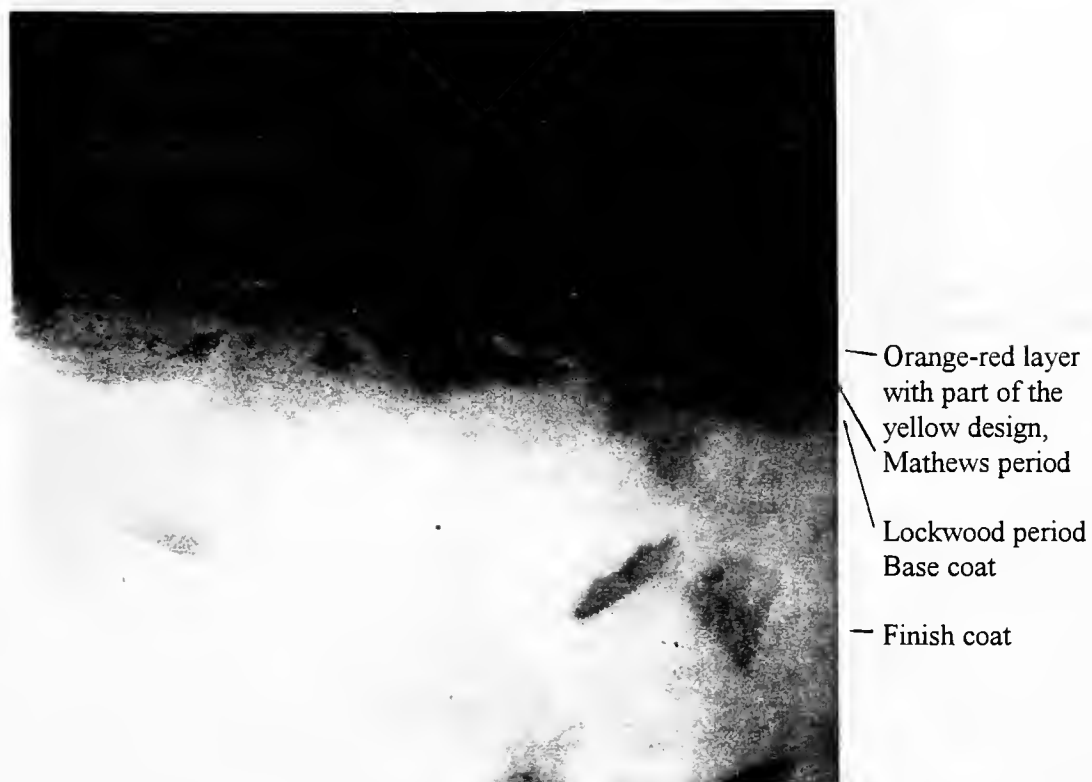
**Location:** Fragments from wall, Mathews period

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 22A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**







**Figure 160: Photo Micrograph of Sample 53: Rotunda**

**Location:** Fragments from ceiling

**Type of Film:** 200 ASA Kodak Royal Gold, Film 3 Negative 23A

**Camera:** Nikon

**Magnification:** 125X

**Reflected light**

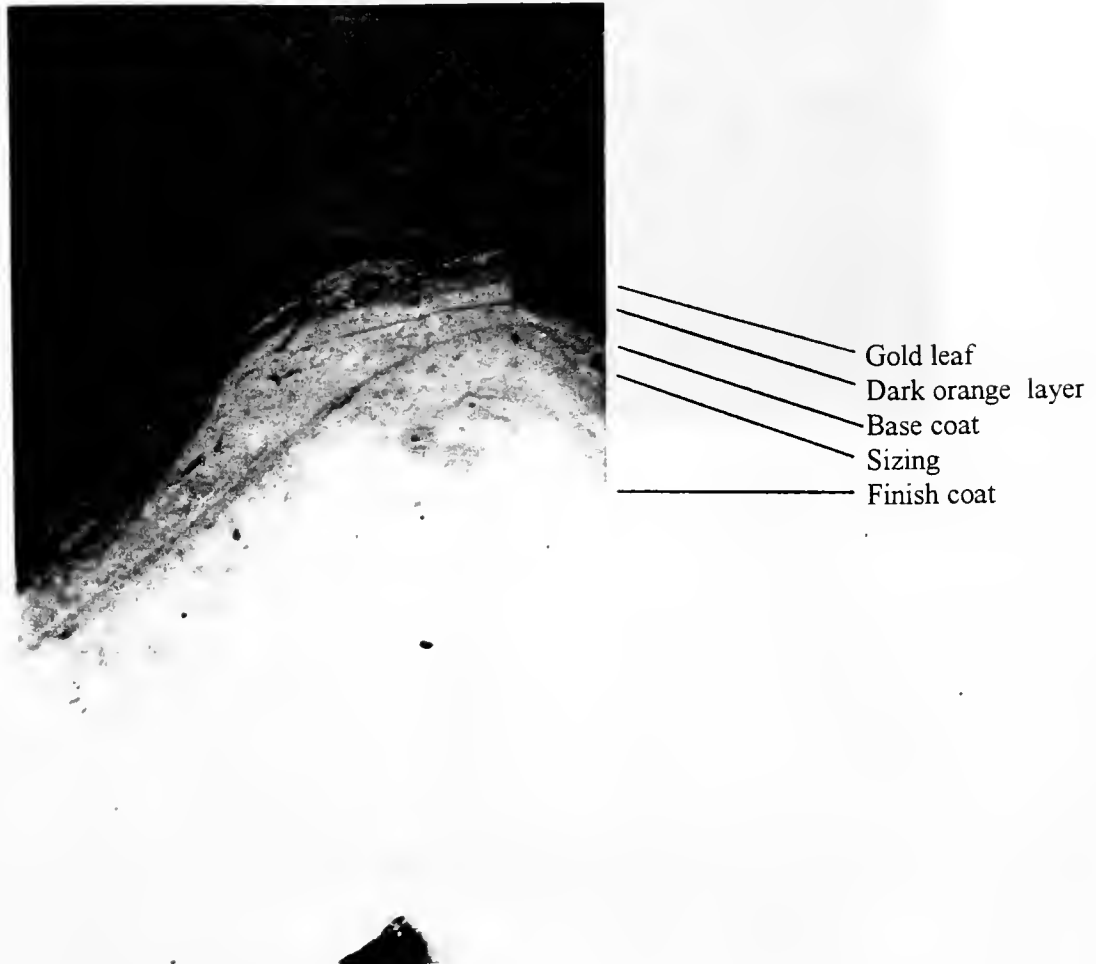
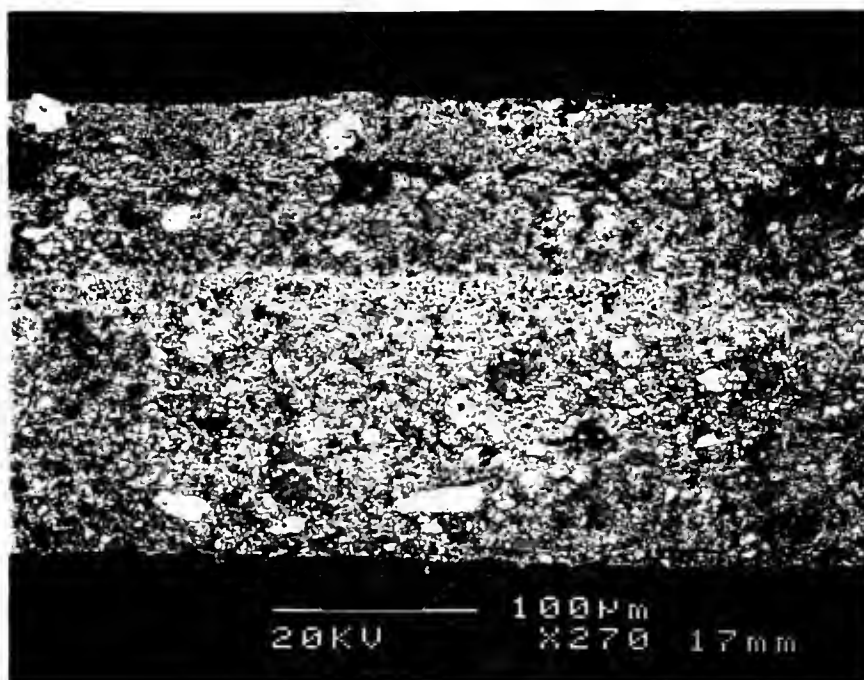




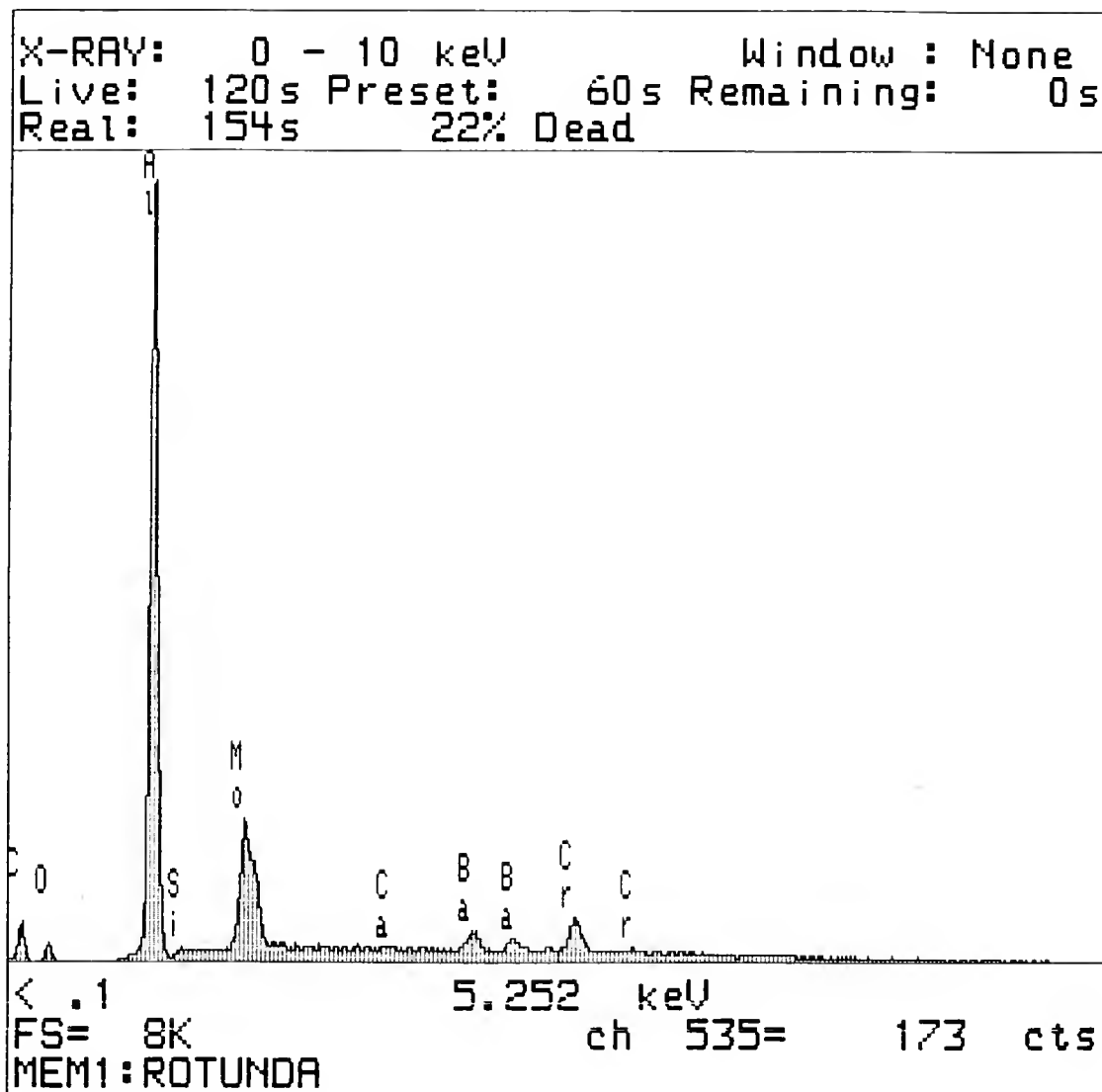
Figure 161: Back Scatter Electron Image of Sample 53: Rotunda





**Figure 162: X-Ray Energy Dispersive Analysis of Sample 53: Rotunda**

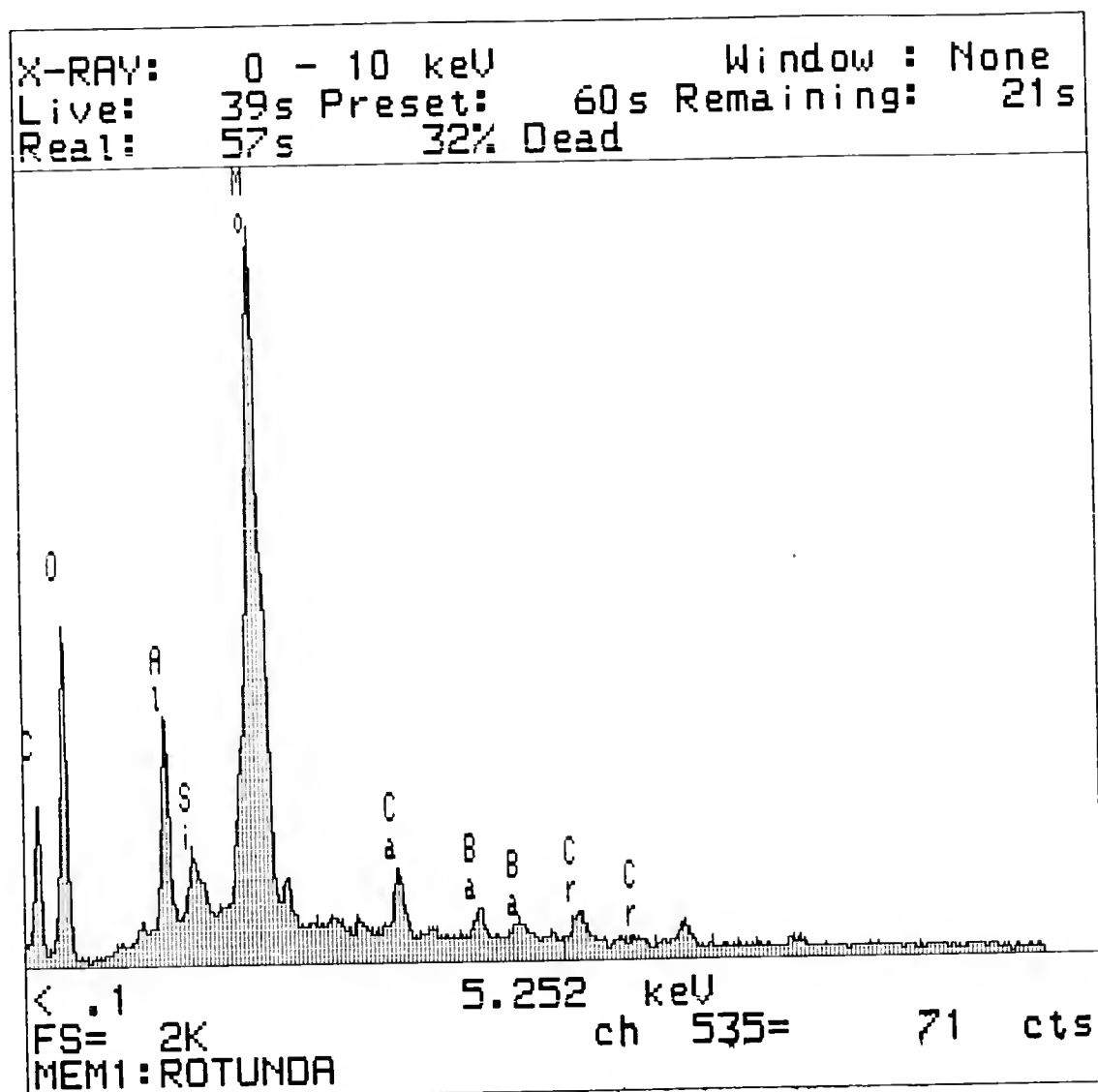
This gold coated sample revealed that the gold leaf was in fact aluminium leaf. Mo and Cr are also present.





**Figure 163: X-Ray Energy Dispersive Analysis of Sample 53: Rotunda**

Sample is from the brown-yellow area. This area of the sample indicates that the substrate of the aluminum leaf consists mainly of molybdates.







**Figure 164: X-Ray Energy Dispersive Analysis of Sample 29: Rotunda**

This carbon coated sample of the Mathews red indicates that vermillion was used.

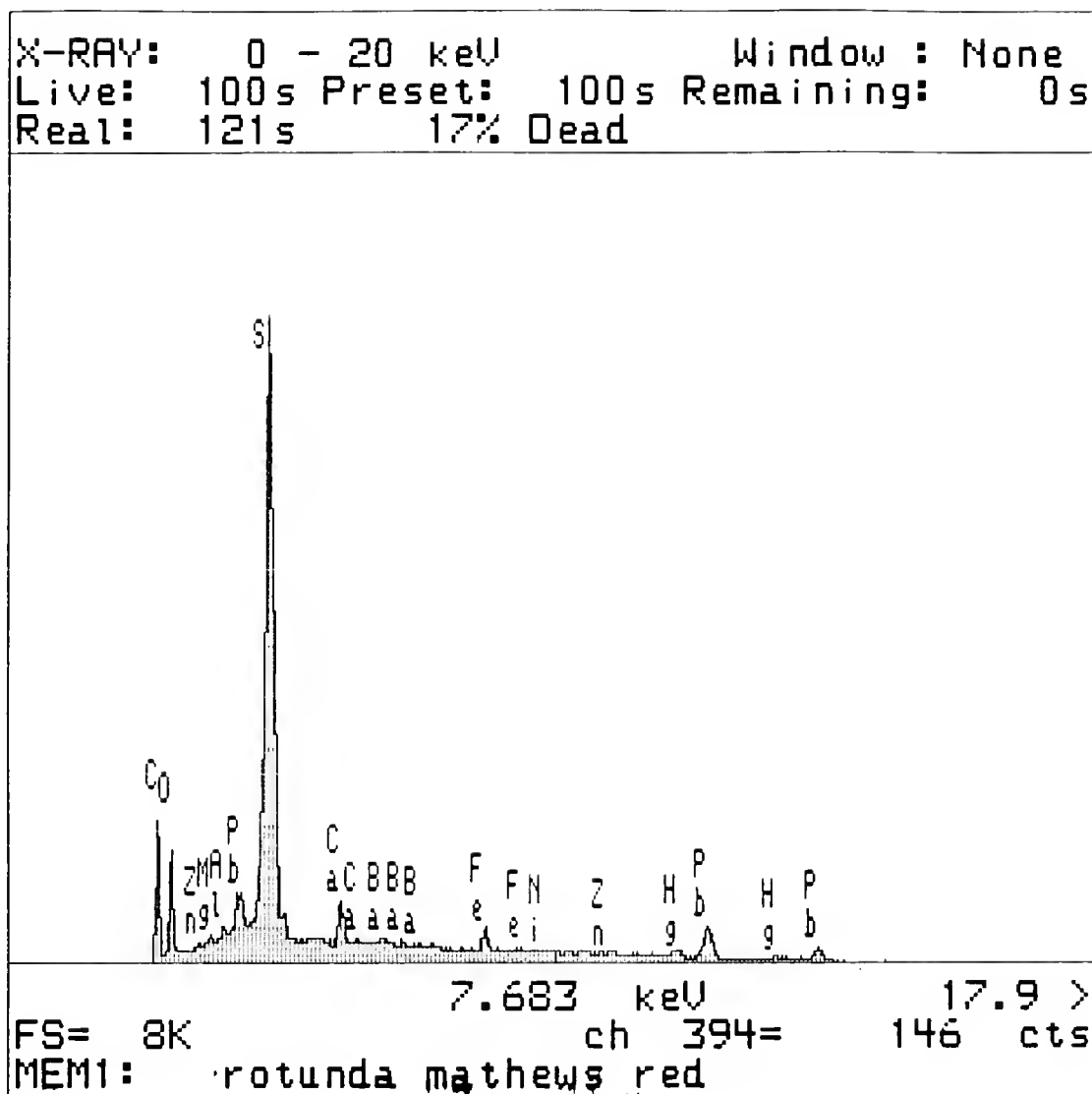
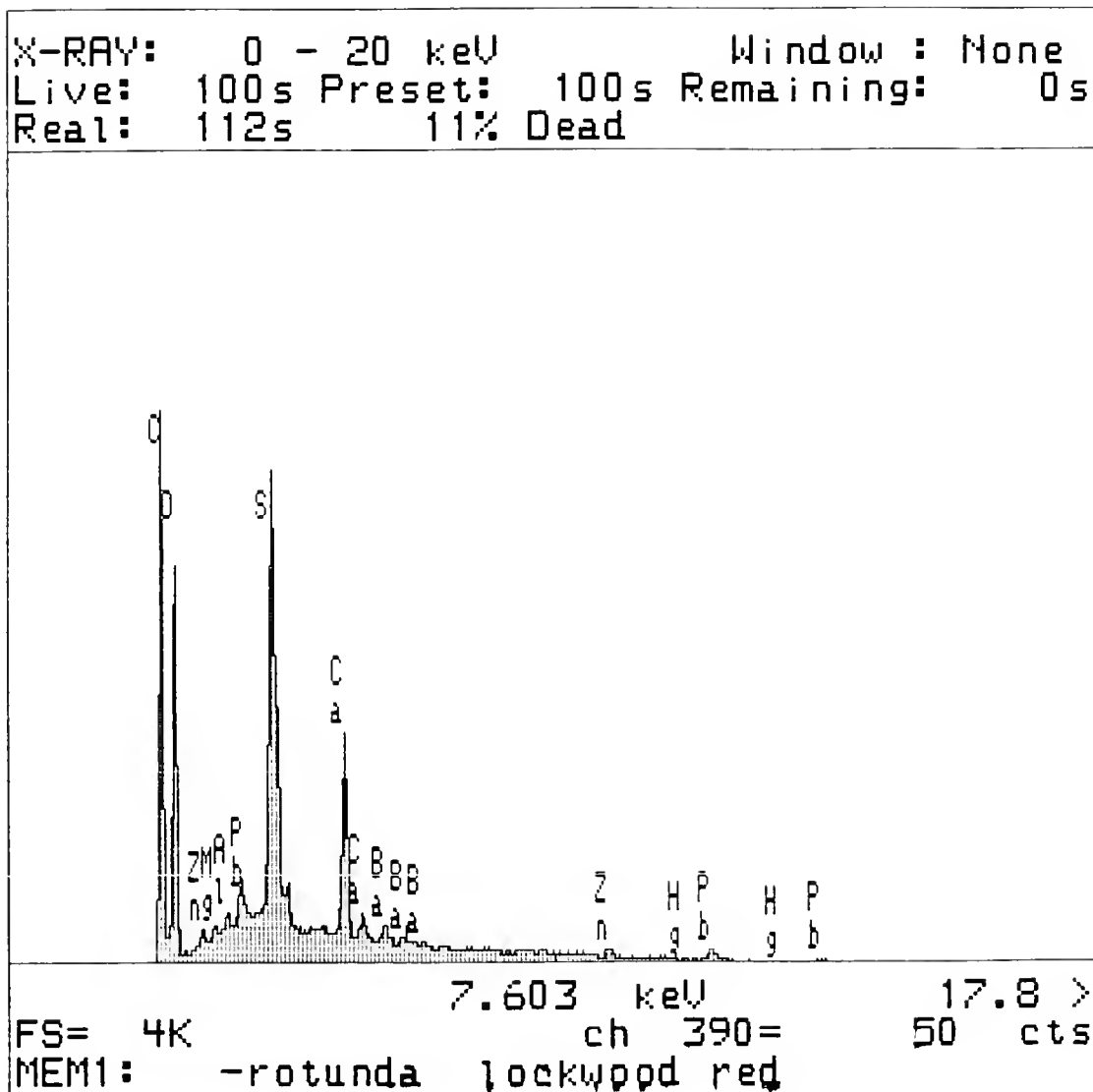




Figure 165: X-Ray Energy Dispersive Analysis of Sample 29: Rotunda

This carbon coated sample of the Lockwood red indicates that an organic red pigment was used, presumably alizarin crimson. It is possible that there is some ultramarine and carbon black in it as well.





## APPENDIX K: MIRROR ROOMS

Figure 166: Back Scatter Electron Image of Sample 62: Mirror Rooms

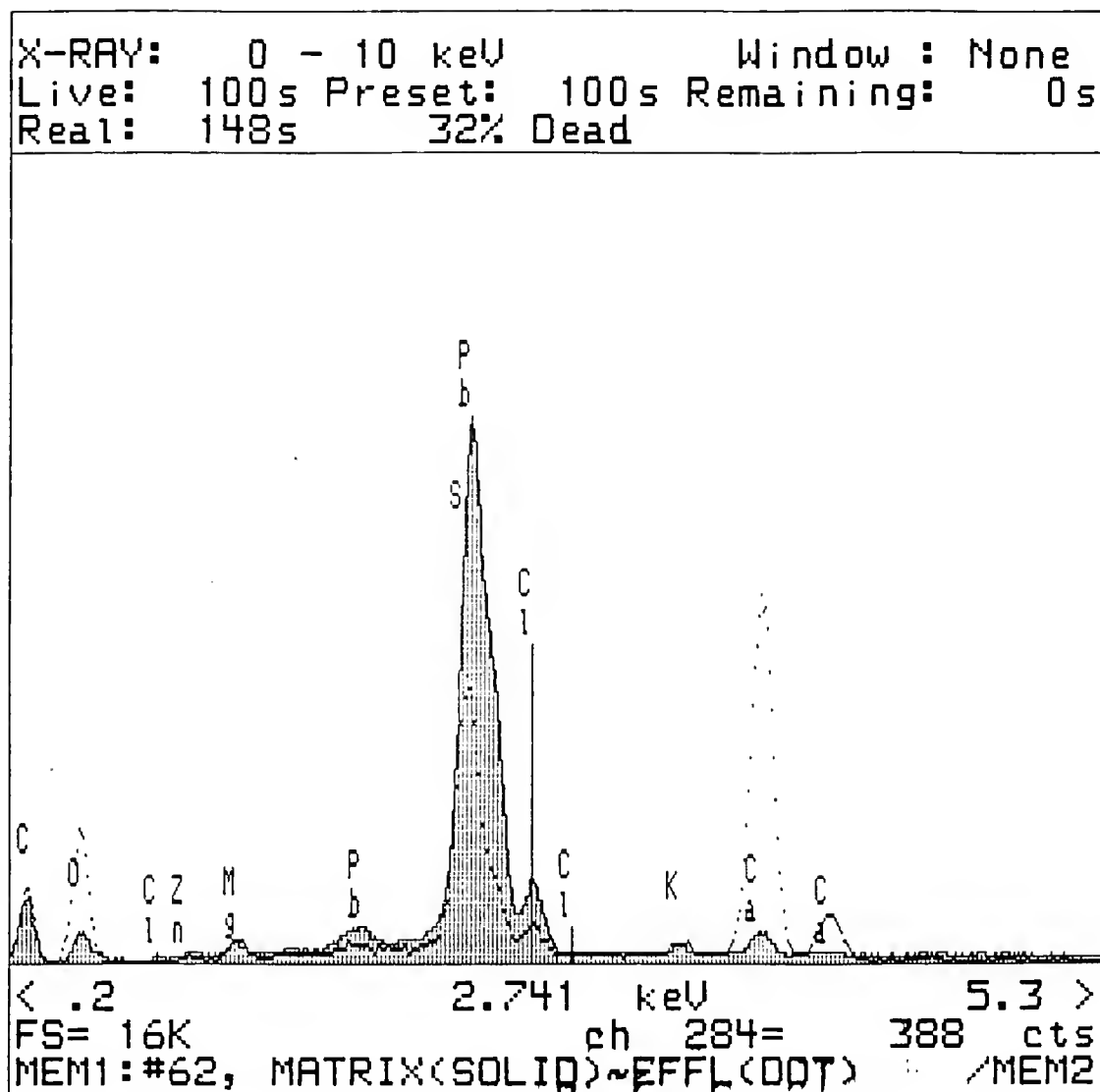




Figure 167: X-Ray Energy Dispersive Analysis of Sample 62: Mirror Rooms

Lead sulfate, lead white and an organic pigment are present in this carbon coated sample.

The sample was removed from field; the color is beige-green.

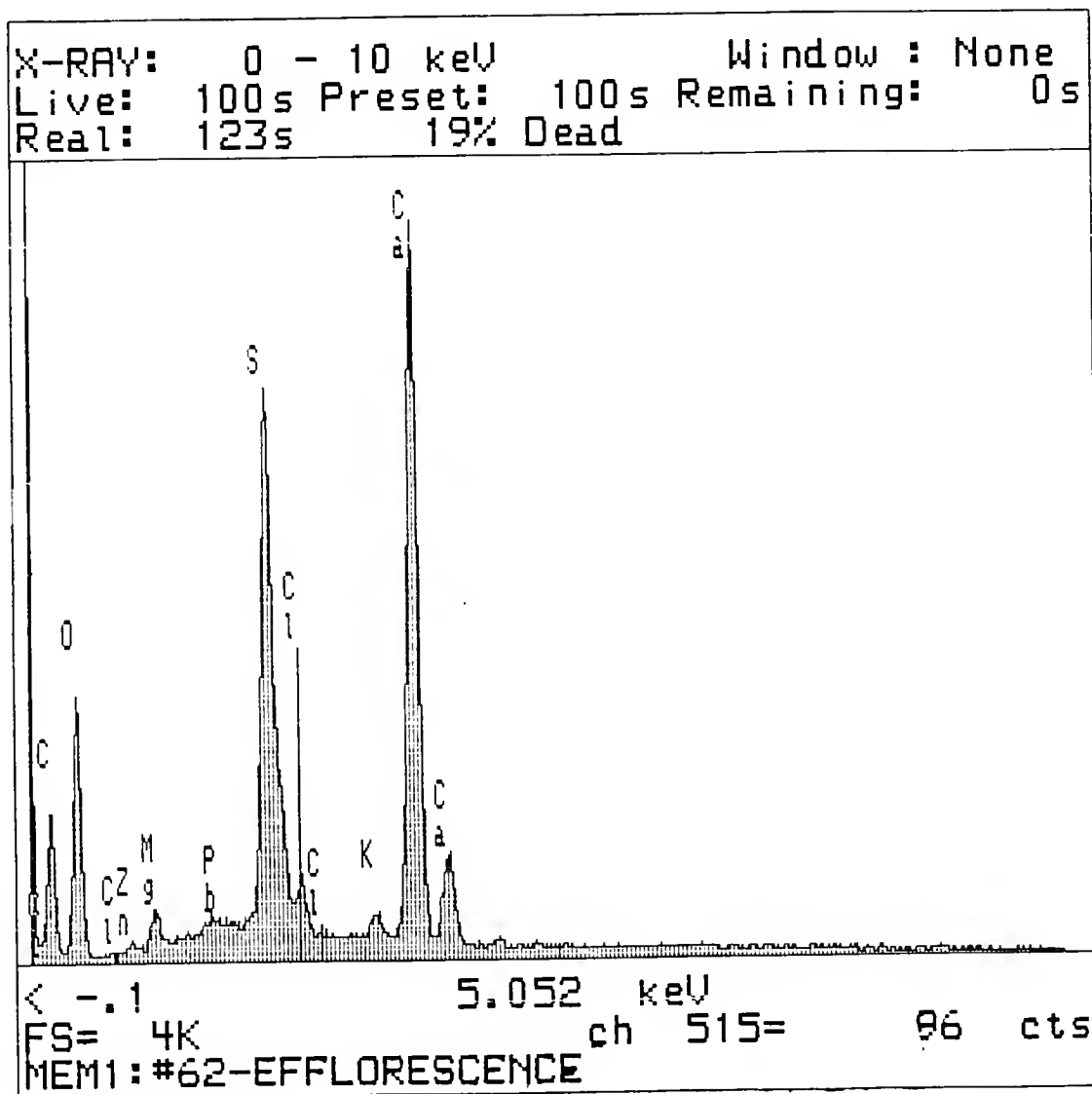






**Figure 168: X-Ray Energy Dispersive Analysis of Sample 62: Mirror Rooms**

Comparison between light and dark areas of the BEI. It indicates that efflorescence took place.





## APPENDIX L: INSTRUMENTS

1. FT-IR: Fourier Transform Infrared Spectrometer with Microscope.

Instrument: Nicolet 510P FT-IR

2. SEM: Scanning Electron Microscopy. (EDAX) Instrument: Joel 64,000

3. XRD: X-Ray Diffraction. LRSM

4. GC: Gas Chromatography. Instrument: Eager 200 Carbon Nitrogen Analyzer

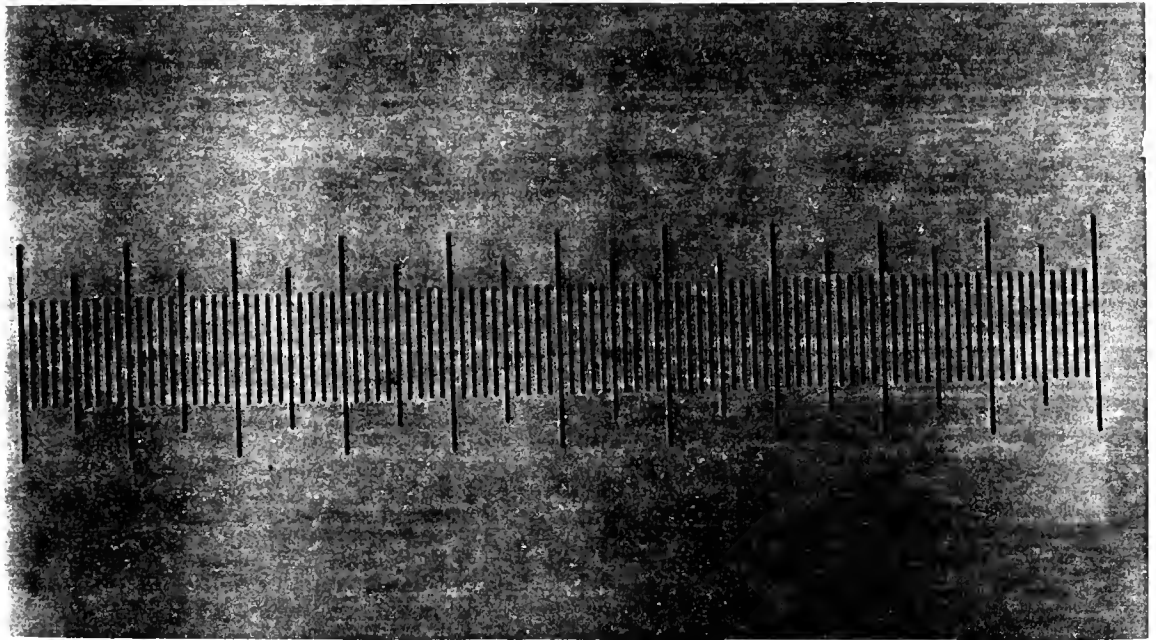
5. Microscopy: Instrument: Nikon Optiphot 2 Polarized Microscope using reflected visible and polarized light. Source: Quartz - Halogen Fiber Optics



# **Figure 169: Photograph Scales**

Photograph scale is 0.01 mm

Magnification is 225X



Magnification is 125X





# APPENDIX M: LIST OF SAMPLES (Phillips)

Slides of Morgan Phillips			Box 1
Room	Number	Specifics	Slide No.
Music	104/A1	green leaf	1
Music	104/A2	green leaf	2
Music	104/B1	beige stripe	3
Music	104/B2	beige stripe	4
Music	104/C1	lavender stripe; <i>not attached</i>	5
Music	104/C2	lavender on blue stripe	6
Music	104/C3	lavender stripe (scrapped)	7
Music	104/C4	lavender stripe (formula 409)	8
Music	104/D1	wall outside; stencil; <i>specimen missing</i>	9
Music	104/D2	wall outside; stencil	10
Music	104/D3	wall outside; stencil	11
Music	104/D4	wall outside; stencil; cleaned; in shadow	12
Music	104/D5	wall outside; stencil; cleaned; in shadow	13
Music	104/E	gold (unscrapped)	14
Music	104/F	strip b/t blue and gold stripes; 5 YR 8/1; <i>falling off slide</i>	15
Music	104/G	inner blue strip	16
Music	104/H	strip b/t blue and little gold; 5 YR 8/1	17
Music	104/I	thin gold stripe	18
Music	104/J1	inside panel	19
Music	104/J2	inside panel	20
Music	104/J3	inside panel; cleaned; shadow; *	21
Music	104/K	orange leaves; 5 YR 6/6	22
Music	104/K3	orange square (ammonia)	23
Music	104/L1	7.5 R 5/6	24
Music	104/M1	2.5 YR 4/8; rosette	25
Music	104/B3K2L2		26
Drawing	105/A, C, D		27
Drawing	105/A1	wall panel	28





Drawing	105/A2	wall (scraped)	29
Drawing	105/A3	walls (cleaned with ammonia); two chunks are off slide	30
Drawing	105/A4	walls (scraped); 5 YR 7/2; specimen missing	31
Drawing	105/B1	orange stripe; 5 YR 6/6	32
Drawing	105/B2	orange stripe; 5 YR 6/6	33
Drawing	105/C1	light stripe; 5 YR 0/1	34
Drawing	105/C2	light stripe	35
Drawing	105/D1	dark leaf; 10 R 7/1	36
Drawing	105/D2	dark leaf and highlight	37
Drawing	105/E	highlight in flower; N8.5	38
Drawing	105/E	highlight in leaf; N8.5	39
Drawing	105/F	flower; 10 YR 7/6	40
Drawing	105/G	flower center; 5 YR 6/6	41
Rotunda	108/A	walls-beige; 10 YR 8/1.5	42
Rotunda	108/B	stencil background; 10 YR 7/2 or 7/4; not attached	43
Rotunda	108/C	leaf-vermillion; 7.5 R 5/12	44
Rotunda	108/D	stripe and outline; specimen missing	45
Rotunda	108/E	flower (gold); specimen not attached	46
Rotunda	108/F	stripe; 10 YR 8/1.5	47
Rotunda	108/G	(b/t stripes); 10 YR 8/1.5	48
Rotunda	108/H	(outside stencil); same as A; 10 YR 8/1	49
Rotunda	cove ceiling	cove rotunda after removal; L: topside; R: underside	50
Rotunda	cove	(Mathews); wall, stencil and shading	51
Rotunda	102/A1, A2	ceiling; grey	52
Entrance Hall	102/A3	grey; piece of specimen not attached	53
Entrance Hall	102/B	ceiling; stripe; specimen not attached	54
Entrance Hall	102/B2	ceiling; blue stripe; (cleaned on left); munsell given, unreadable	55
Entrance Hall	102/C	ceiling; moldings	56
Entrance Hall	102/D1D2D3	wall; 10 YR 7/1.5	57
Entrance Hall	102/E	niche frame; 5 B 7/2	58
Entrance Hall		Hall b/t Entrance Hall and Rotunda; 10 YR 7.5/1; not attached	59
Hall		beige; over door; 101 4R 7/2	60
Vestibule	101/A		61



Vestibule	101/B	beige; over door; 104 R 8/1.2	62
Vestibule	101/C1,2,3	coral rectangle; 7.5 R 5.5/8	63
Vestibule	101/D	blue circle; 10 B 7/2	64
Vestibule	101/E	coral rectangle; 7.5 R 5.5/8	65
Vestibule	101/F	beige; framework	66
Vestibule	101/G1, G2	beige; convex molds; window	67
Vestibule	101/H1,2,3,4	stencil; stripe	68
Vestibule	101/I	beige wall (plaster); 10 YR 7/2	69
Vestibule	101/J	small convex molding scraped; too FRR?; 10 YR 8/2	70
Vestibule	101/J2	small convex molding flaked; 10 YR 7/1.5; <i>not attached</i>	71
Vestibule	101/K	cema?; molding; flaked; 10 YR 7/1.5	72
Vestibule	101/L	flat frame; molding	73
Vestibule	101/M2	big convex molding (son); 10 YR 8/2	74
Vestibule	101/N	Big convex molding (door); 10 R 5/10, <i>currently in box 2</i>	75
Library	103/A	ceiling; blue	76
Library	103/B	ceiling; brown	77
Library	103/C	ceiling; white, grey	78
Library	103/D	ceiling; dark blue	79
Library	103/E	ceiling; black	80
Library	103/F	ceiling; coral	81
Library	103/G	ceiling; rose; et. cleaned?	82
Library	103/H	(cleaned)	83
Library	103/I, J	I: gold; J: black	84
Library	103/K	(cleaned); 7.5 R 5/12; <i>specimen not attached</i>	85
Library	103/L	border leaf; (cleaned)	86
Library	103/M	border diamond; (cleaned)	87
Library	103/N, O	N: green diamond; O: yellow stripe; (cleaned)	88
Library	103/P	"grey" background	89
Library	103/Q	<i>specimens not attached</i>	90
Library	103/R	Palmette	91



Slides of Morgan Phillips			Box 2
Room	Number	Specifics	Slide No.
S-R-F	1	Right: Bottom Up; Left: Top Up	1
S-R-F	2		2
S-R-F	3	A/Left: Top Up; A/Right: Bottom Up; B: Bottom Up	3
S-R-F	4		4
S-R-F	5		5
S-R-F	6	Left: Bottom Up; Right: Top Up	6
S-R-F	7	Left: Top Up; Center and Right: Bottom Up	7
S-R-F	8	Left: Bottom Up; Center and Right: Top Up	8
Rotunda	(Matthews)	"Greekfret"?; 5 YR 7/6	9
Rotunda	(Matthews)	Whil?; 10 R 4/8; <i>part of specimen not attached</i>	10
Roof		wood 1/4 rosette	11
Roof		wood det.; roof crocket A, B, C	12
Top of Gables		wood cornice; new primer; <i>specimen not attached</i>	13
?		window frame or palmette on skylight	14
Fourth Floor		cast iron windows	15
Iron Palmette		10 inches; (upside down chip)	16
Billard Room	109/A	wall; inside border; <i>specimen not attached</i>	17
Billard Room	109/B	gold; <i>specimen missing</i>	18
Billard Room	109/C	beige stripe	19
Billard Room	109/D	shading to brown stripe	20
Billard Room	109/E	blue stripe; 2.5 PB 7/2	21
Billard Room	109/F & G	shading to blue stripe; 5 PB 4/4	22
Billard Room	109/G	border background; 2.5 Y 8/4	23
Billard Room	109/H	leaf and rosette; N3.5	24
Billard Room	109/I	wall; outside border; 5 Y 7/1	25
Billard Room	109	City of Norwalk; all walls; 7.5 B, 5 B 8/4	26
Hallway	110/A	wall; 10 Y 9/1	27
Hallway	110/B	gold; <i>specimen missing</i>	28



Hallway	110/C	light brown stripe; 7.5 YR 5/6	29
Hallway	110/D	dark brown; 7.5 YR 5/4	30
Hallway	110/E	interlacing; 7.5 YR 7/4	31
Hallway	110/F	blue stone; N3.5	32
Hallway	110/G	blue; 5 B 7/1	33
Hallway	110	<i>big specimen not attached</i>	34
Dining	107/A1	wall; green; exposed to sun; 10 Y 7/1; <i>not attached</i>	35
Dining	107/A, E	over west door	36
Dining	107/A2, C2	wall; behind west cornice	37
Dining	107/B	gold; specimen missing	38
Dining	107/C	green stripe and leaf shading; <i>not attached</i>	39
<i>There are no Dining 107/D, E, F or I in the box</i>			40
Dining	107/G	7.5 YR 8/2	41
Dining	107/H	shading to G and stripe; 7.5 YR 5/4	42
Dining	107/J	cornice crown mold; dark stripe; 7.5 YR 3/2	43
Breakfast		wall; 10 YR 7/2	44





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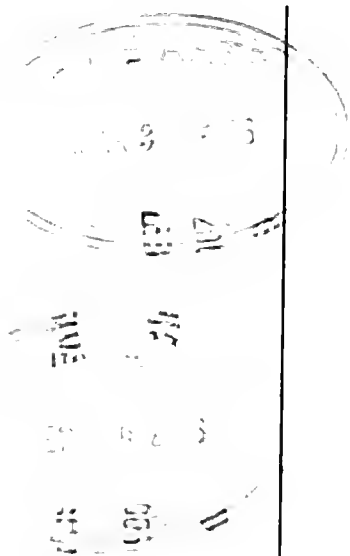


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